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## Summary Report for Grant NAG/1/1476

Engineering Design of Sub-Micron Topographies for  
Simultaneously Adherent and Reflective Metal-Polymer Interfaces

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(NASA-CR-194573) ENGINEERING  
DESIGN OF SUB-MICRON TOPOGRAPHIES  
FOR SIMULTANEOUSLY ADHERENT AND  
REFLECTIVE METAL-POLYMER INTERFACES  
(Worcester Polytechnic Inst.) 56 p

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## **INTRODUCTION**

The approach of the project is to base the design of multi-function, reflective topographies on the theory that topographically dependent phenomena react with surfaces and interfaces at certain scales. The first phase of the project emphasizes the development of methods for understanding the sizes of topographic features which influence reflectivity. Subsequent phases, if necessary, will address the scales of interaction for adhesion and manufacturing processes.

A simulation of the interaction of electromagnetic radiation, or light, with a reflective surface is performed using specialized software. Reflectivity of the surface as a function of scale is evaluated and the results from the simulation are compared with reflectivity measurements made on multi-function, reflective surfaces.

## **METHODS**

### **Simulation**

In this work a numerical simulation of light interaction with a surface is compared with reflectivity measurements made at NASA with a Perkin-Elmer Lambda5 scatterometer. Light is emitted from a source, reflected by a surface and then intersects a detector.

### **Topography**

In the simulation, we represented the topography with a 200 x 200 grid of points acquired by a scanning tunneling microscope (STM), where the points are located in a grid in x and y with a height z. The STM work was performed at NASA Langley on a Digital Equipment Nanoscope II. Six topographies were scanned at three scan sizes, 20 $\mu$ m x 20 $\mu$ m, 2 $\mu$ m x 2 $\mu$ m, and 200nm x 200nm (see Table 1).

The large, topographic data sets, used to represent the surface, are analyzed by the patchwork method where the surface, represented by the data points, is tiled with triangular patches (Brown et al. 1992). The topography is evaluated over a range of scales, or patch sizes, by tiling over the surface with decreasing patch sizes. In the simulation each patch represents a reflective facet, atomically smooth and a perfect reflector.

The triangular patches are placed on the surface in two directions: parallel and perpendicular to the STM scan direction. Reflectivity calculations are made for each direction and the results of the simulation are the average of the two calculations.

### **Lambda5 Scatterometer**

We created a computer model of the Perkin-Elmer Lambda5 scatterometer from optical path representations of the scatterometer and reflectivity assembly which were provided by Perkin-Elmer. The incident angle of the light is user-defined, and the detector was modeled as a rectangle at the position and orientation defined by the optical schematic. The size of detector was defined from an engineering drawing of the detector which was

provided by Hammamatsu (part number R298 HA). The output of the detector was assigned a value of unity for any intersection of a reflected ray with the detector.

**Table 1 STM Scan and Reflectivity Measurements**

SURFACE	STM FILENAME	MATERIAL	POINT SPACING X x Y x Z (nm)	REFLECT % 20 DEGREES 531 nm
A	NASA22	T7M1R-P1	100	69.1
A	NASA23	T7M1R-P1	10	69.1
A	NASA24	T7M1R-P1	1	69.1
B	NASA25	P4H1R	100	46.7
B	NASA26	P4H1R	10	46.7
B	NASA27	P4H1R	1	46.7
C	NASA16	D25M2R-P30	100	44.7
C	NASA17	D25M2R-P30	10	44.7
C	NASA18	D25M2R-P30	1	44.7
D	NASA19	T7H3R	100	44.1
D	NASA20	T7H3R	10	44.1
D	NASA21	T7H3R	1	44.1
E	NASA13	D25H1R-P30	100	15.2
E	NASA14	D25H1R-P30	10	15.2
E	NASA15	D25H1R-P30	1	15.2
F	NASA10	P4M1R	100	14.1
F	NASA11	P4M1R	10	14.1
F	NASA12	P4M1R	1	14.1

The light emitted by the source was modeled as set of parallel rays that originate from the source, travel to the surface and are reflected by the center of each patch. One ray is generated for each patch. The direction of the reflected ray is calculated from the incident ray direction and the normal of the patch. From the center of the patch, the ray is reflected off of an optical wedge, a concave, spherical mirror and then it is determined if the ray intersects the detector.

The output of the Lambda5 is percent reflection relative to a known reference sample, in this case the reference sample was a stainless steel mirror provided by Perkin-Elmer. The reflectivity measurements are expressed in terms of a percentage of the measured reflectance from the reference sample.

#### **Reflectivity Simulation**

We ran the computerized simulation on the 18 STM data sets with an incident angle of 20 degrees. The simulation generated the incident rays, reflected them off the patches and counted the rays which intersected the detector. The output of the simulation  $R_r$  or absolute percent reflectivity, is defined as the number of incident rays that intersect the

detector is divided by the total number of rays reflected by the surface.  $R$  is calculated for each patch size and is plotted versus  $\log(\text{patch area})$ .  $R_R$ , or relative reflectivity, is calculated from  $R$  as

$$R_{RA-B} = (RA-RB)/RA \quad [\text{eq. 1}]$$

where  $RA > RB$ .

The simulation was modified to account for the effect of patch orientation on the intensity of the incident rays. The intensity of the incident ray, initially equal to one, is multiplied by the cosine of the angle between the patch normal and the incident ray. The ray is then reflected by the patch and the simulation records the number of rays and their intensities that intersect the detector. The output of the weighted reflectivity simulation,  $R_w$ , is

$$R_w = (\Sigma \text{ intensity collected rays}) / \text{number of reflected rays.} \quad [\text{eq. 2}]$$

### Reflectivity Results

We combined the simulation's reflectivity results from the three scan sizes into one larger set of results using Matlab, a matrix-based software program. The large set of results, combined for each of the six surfaces, covered a range of patch areas of 7 orders of magnitude, from  $0.5 \text{ nm}^2$  to  $3 \mu\text{m}^2$ . Table 2 shows the range of scale for each of the three scan sizes and the data point spacing of the scans.

**Table 2. Large and Small Patch Sizes for Three STM Scan Sizes**

Scan Size	Large Patch Size	Small Patch Size	Point Spacing
20 $\mu\text{m}$ x 20 $\mu\text{m}$	3 $\mu\text{m}^2$	5000 $\text{nm}^2$	100 $\text{nm}$
2 $\mu\text{m}$ x 2 $\mu\text{m}$	5000 $\text{nm}^2$	50 $\text{nm}^2$	10 $\text{nm}$
200 $\text{nm}$ x 200 $\text{nm}$	50 $\text{nm}^2$	0.5 $\text{nm}^2$	1 $\text{nm}$

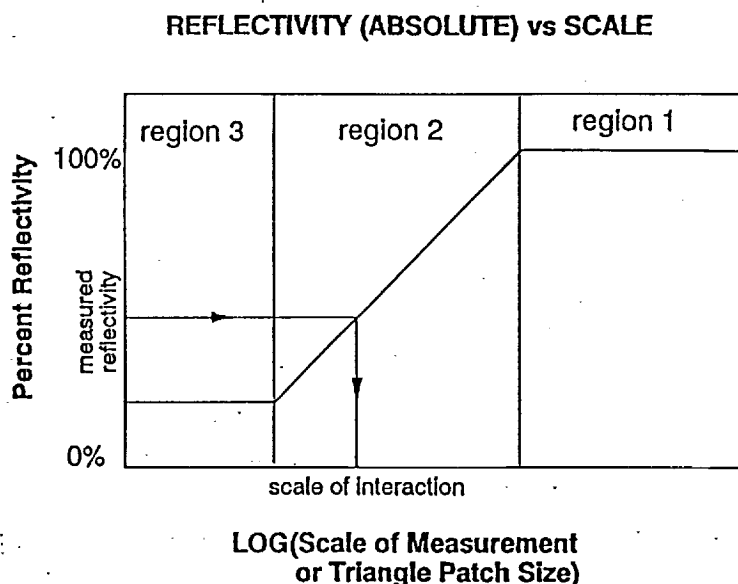
When the scan sizes were combined, the magnitudes of  $R$  did not correspond at the joining patch size. The values of  $R$  were shifted by the difference between the scans so as to match at the joining patch size. The  $R$  values of the largest scan size (20 $\mu\text{m}$  x 20 $\mu\text{m}$ ) were used as the zero shift scan when the scans were shifted up, and the  $R$  values of the two scans (2 $\mu\text{m}$  x 2 $\mu\text{m}$  and 200 $\text{nm}$  x 200 $\text{nm}$ ) were shifted up to the zero shift scan. When the scans were shifted down, the smallest scan size (200 $\text{nm}$  x 200 $\text{nm}$ ) was the zero shift scan, and the two larger scans were shifted down to it. The shifted results were used to generate plots of absolute reflectivity vs.  $\log(\text{patch area})$ .

Relative reflectivity results were calculated from the shifted scans for shifted up results, and plots of relative reflectivity vs.  $\log(\text{patch area})$  were generated. Calculation of relative reflectivity ( $R_R$ ), from eq. 1, was designed to factor out the dependence on the reference sample. Surface A, which has the largest reflectivity measurement, was used as the

reference in the plots. A negative value of  $R_R$  indicates that the reflectivity of the surface is greater than surface A.

### Scale of Interaction

The results of the reflectivity simulation are compared to experimental results, obtained from NASA Langley's Lambda5, to calculate a scale of interaction of the light with the surface. The scale of interaction was defined as the square root of the patch area, from the reflectivity simulation, where the corresponding magnitude of  $R$  is equal to the reflectivity value measured by NASA on the Lambda5. Figure 1 shows how a scale of interaction is found from the simulation and experimental results.



**Figure 1- Schematic of Calculation of Scale of Interaction from Idealized, Absolute Reflectivity vs. Scale** The intersection of the measured reflectivity is found, and the scale of interaction is calculated from the corresponding patch size. The scale of interaction is shown to occur in region 2.

## RESULTS

### Relative Area

The STM data sets were analyzed by the patchwork method and a representative scale-area plot is shown in figure 2 (Brown et al. 1993). The scale-area analyses were conducted on each of the three scan sizes separately. All of the scale-area plots are found in Appendix A.

### Absolute Reflectivity

Absolute reflectivity as a function of scale, or patch area, was calculated by the reflectivity simulation. Shifted up and shifted down results for surface A are shown in Figures 3 and 4. Absolute reflectivity plots for the six surfaces are found in Appendix B.

### Cosine Weighting - incident angle

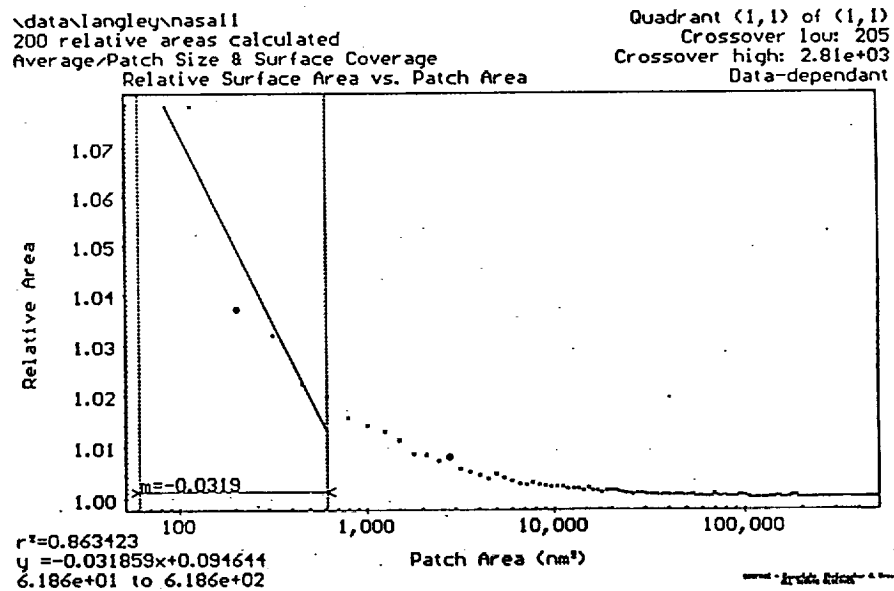
Absolute, cosine-weighted reflectivity as a function of scale, for surface B, is plotted with non-weighted results in Figure 5. Cosine-weighted results for the six surfaces are found in Appendix C.

### Relative Reflectivity

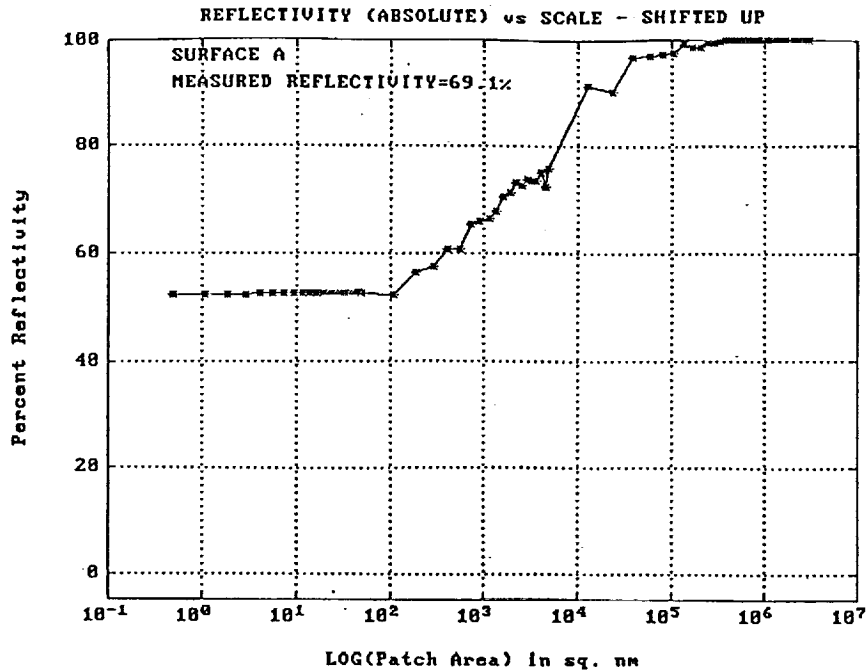
Relative reflectivity as a function of scale, for surface B relative to A, is shown in figure 5. Relative reflectivity plots for surfaces B through F, relative to A, were generated using shifted up results. The plots are found in Appendix C.

### Scale of Interaction

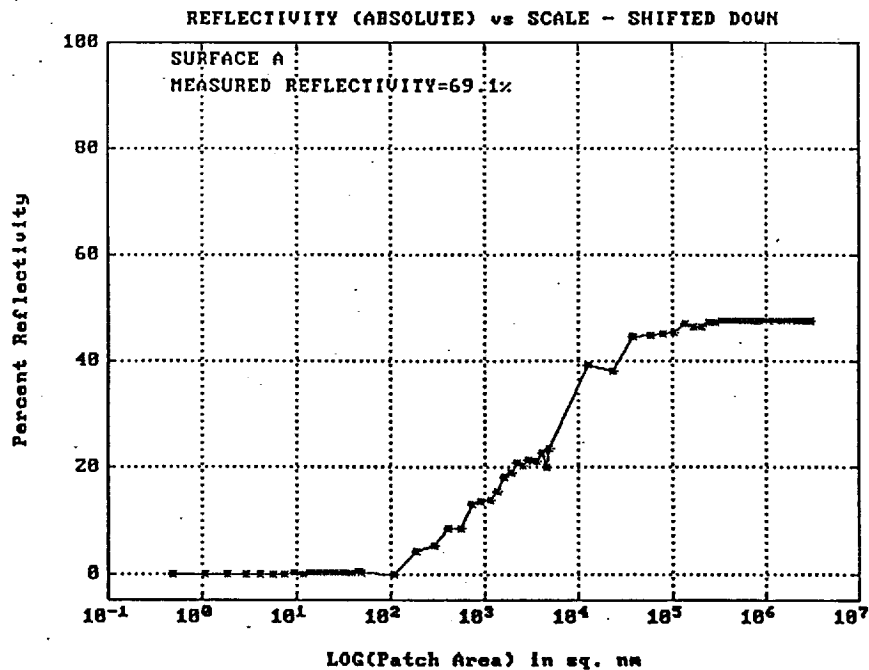
A scale of interaction was calculated from the absolute reflectivity plots. Table 3 lists the scales of interaction found using the absolute reflectivity results. The reflectivity measurements did not intersect the relative reflectivity results, and no scales of interaction were found.



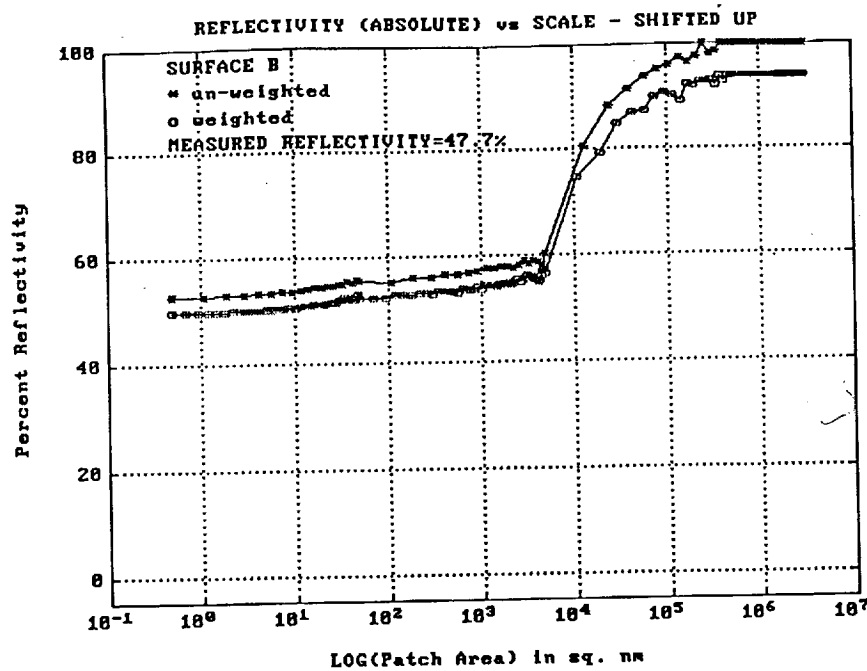
**Figure 2 - Scale-Area Plot of Surface A.** The 2mm x 2mm scan size is shown for surface A. The relative area begins to increase (crossover) at a patch area of 2810 nm².



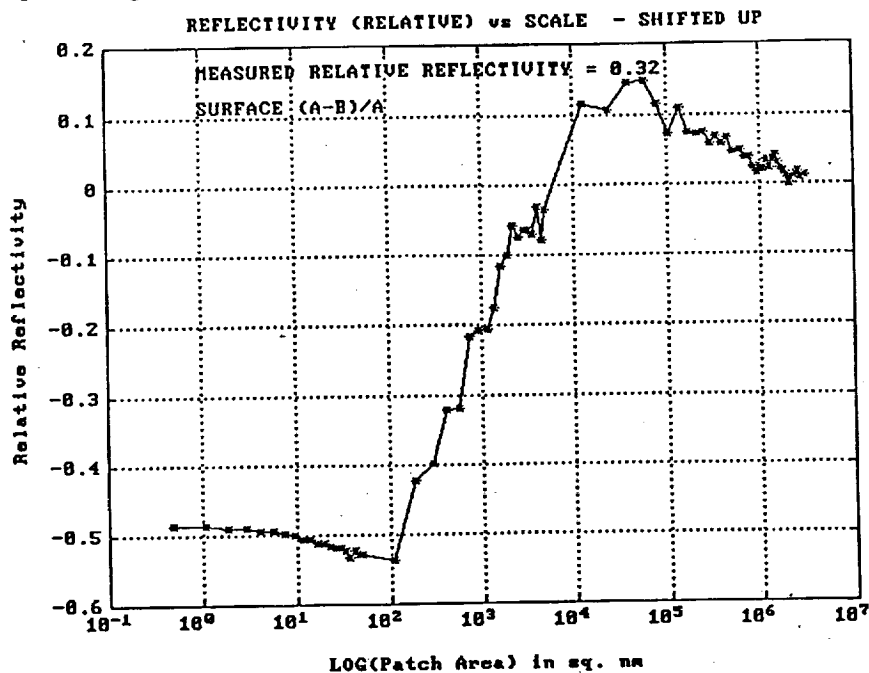
**Figure 3 - Shifted Up Absolute Reflectivity Results for Surface A** The percent reflectivity is 100% at large patch areas and decreases to about 53% at the fine patch sizes. The largest scan size was used as the no shift scan.



**Figure 4 - Shifted Down Absolute Reflectivity Results for Surface A.** The percent reflectivity is about 48% at large patch areas and decreases to about 0% at the fine patch sizes. The smallest scan size was used as the no shift scan.



**Figure 5 - Incident Angle Cosine-Weighted and Non-Weighted Absolute Reflectivity Results for Surface B.** The difference between the cosine weighted and unweighted results is shown. Cosine weighting shifts the set of results down by a factor of 0.94 (a multiplier of  $\cos 20^\circ$ ), or shifting the results down by 6%. The results are shifted by an equal percentage at all scales.



**Figure 6 - Relative Reflectivity Results for Surface B Relative to Surface A.** The maximum relative reflectivity value is about .15 which is less than the measured result.  $R_R$  is negative at patch areas less than about 500 nm<sup>2</sup>.



**Table 3 Scales of Interaction from Absolute Reflectivity Results.** Surface A is the surface with a scale of interaction from the shifted up results. The scales from the shifted down differ by about 1 order of magnitude.

Surface	Scale of Interaction (nm) shifted up	Scale of Interaction (nm) shifted down
A	39	x
B	x	x
C	x	102
D	x	164
E	x	13
F	x	18

## DISCUSSION

### Relative Area

The scale-area plots show that the reflective topographies are complex at fine scales, and the relative areas, a measure of complexity, increase with decreasing patch size. The difference between the relative areas of the three scan sizes is clear: the maximum, relative areas of the 200nm x 200nm scan size are approximately 2 orders of magnitude larger than that of the 2  $\mu\text{m}$  x 2 $\mu\text{m}$  scan size and approximately three orders of magnitude larger the smallest scan size.

### Absolute Reflectivity

The plots show three distinct regions over a range of scale. In region 1, occurring at patch sizes down to  $10^5 \text{ nm}^2$ , the percentage reflectivity (R) remains constant over a range of patch sizes. Decreasing patch size does not change the amount of reflected light that reaches the detector, and R is largest in this region.

In region 2, occurring at patch sizes from  $10^5 \text{ nm}^2$  to  $100 \text{ nm}^2$ , R decreases with decreasing patch size. Decreasing patch size decreases the amount of reflected light that reaches the detector, and R in this region is less than region 1 and greater than region 3.

In region 3, occurring from a patch size of  $100 \text{ nm}^2$  to  $0.5 \text{ nm}^2$ , R remains constant. Decreasing patch size no longer decreases the amount of light that reaches the detector, and R is lowest in this region.

The material properties of the surfaces, i.e., conductivity, absorbtivity and transmissivity, were not considered in the simulation, and would shift the plots down from the shifted up condition. The downward shift would increase the calculated scale of interaction, and, depending on the size of the shift, would cause the measured reflectivity results to intersect the simulation's results.

Incident angle cosine weighting shifts the reflectivity simulation results down for all of the surfaces, and the percentage shift is constant for all scales. It is speculated that the percentage shift is constant at all scales because the collector is small, and only rays close to the direction of specular reflection are collected. Reflected rays with a weighting factor close to zero would reduce the results by more than 6%, but these rays, with a small weighting factor, will not be reflected close to the specular direction, and will not intersect the detector. The weighting factors of the collected rays are close to cosine ( $20^\circ$ ), and shift the results down by 6%.

### **Relative Reflectivity**

The measured results do not intersect the relative reflectivity results at any scale, which may be because the scale of interaction theory is wrong or because we are misinterpreting the results of the reflectivity measurements. It was expected that all relative values would be positive, at least at one scale, because the calculations were made relative to the surface with the largest reflectivity measurement, surface A, and that this scale would correlate with the reflectivity measurements. The plots show that surface A is less reflective than most of the other surfaces and that the surface with the largest  $R_R$  changes with scale.

The reflectivity measurements made by NASA are expressed as a percentage reflectivity of a stainless steel reference sample. It is not yet clear how we should interpret this representation of reflectivity compared to the computer simulation. Relative reflectivity plots were generated to factor out the dependence on the reference sample, but we have not been successful. Including conductivity and absorptivity of the reference sample in the computer simulation may provide a truer representation of NASA's reflectivity method. Also, more information about how the Lambda5 processes the output signal from the photo multiplier tube may give a better understanding of the equipment's output.

### **Scale of Interaction**

Joining the three scan sizes effects the scale of interaction calculation; shifting up decreases the scale and shifting down increases it. Possible causes of mismatch at joining patch areas are the patch placement algorithm, differences in the STM scan parameters for the three scan sizes or variability in material properties over the different scan areas.

The current algorithm places a small number of patches at the large scales (large with respect to data point spacing), and may provide a poor representation of the topography. Since the topography may be more precisely represented with decreasing patch size, the joining patch size may be thought of as a boundary separating regions of high and low precision.

Changes in material properties of a surface will also change how the tip interacts with the topography. Efforts were made to minimize the effect of local changes in material properties, but the scans may have been effected to some degree.

### **Future Work**

The work will be continued under a NASA training grant, grant number NGT-51107. In future work other reflectivity methods, such as total integrated scattering (TIS), will be investigated as a means for better understanding the amount of energy reflected by the surfaces. Reflectivity samples will be made from homogeneous materials to reduce the complexity of the reflectivity simulation by eliminating multiple layer materials and distributed reflective particles. Random patch placement algorithms will be investigated that may better represent the interaction of light with the surfaces.

### **CONCLUSIONS**

1. The scale-area plots show that the reflective topographies are complex at fine scales, and that the relative areas, a measure of complexity, increase with decreasing patch size.
2. Simulation of a reflecting surface as a collection of triangular mirrors and decreasing the size on each repetition results in a steady decrease in the amount of light arriving at a detector, indicating increasing scatter or diffuse reflection at finer scales.
3. Ranking of the surfaces based on reflectivity calculated from the current algorithm does not correspond at any scale, to the ranking from reflectivity measurement, as they are currently interpreted.
4. The scales of interaction calculated from the current algorithm do not share a common region of reflectivity with the measured values.

### **REFERENCES**

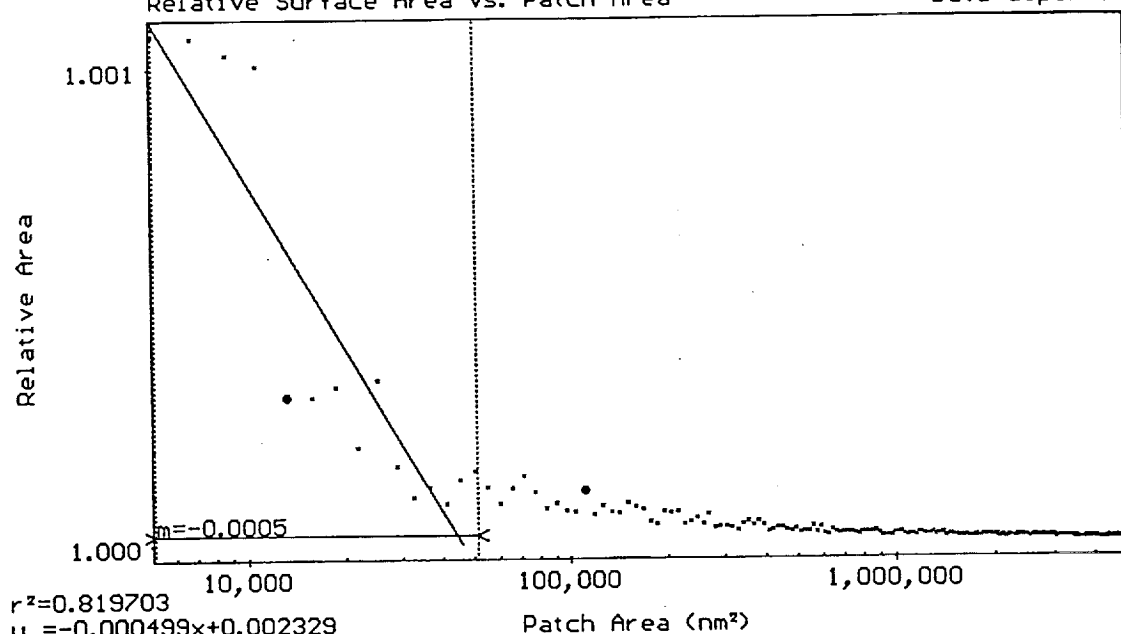
C.A. Brown, P.D. Charles, W.A. Johnsen, S. Chesters, "Fractal analysis of topographic data by the patchwork method", *WEAR*, 161, 61-67, (1993).

## **Appendix A**

### **Scale-Area plots**

\data\langley\nasa10  
 200 relative areas calculated  
 Average/Patch Size & Surface Coverage  
 Relative Surface Area vs. Patch Area

Quadrant (1,1) of (1,1)  
 Crossover low: 1.33e+04  
 Crossover high: 1.11e+05  
 Data-dependant

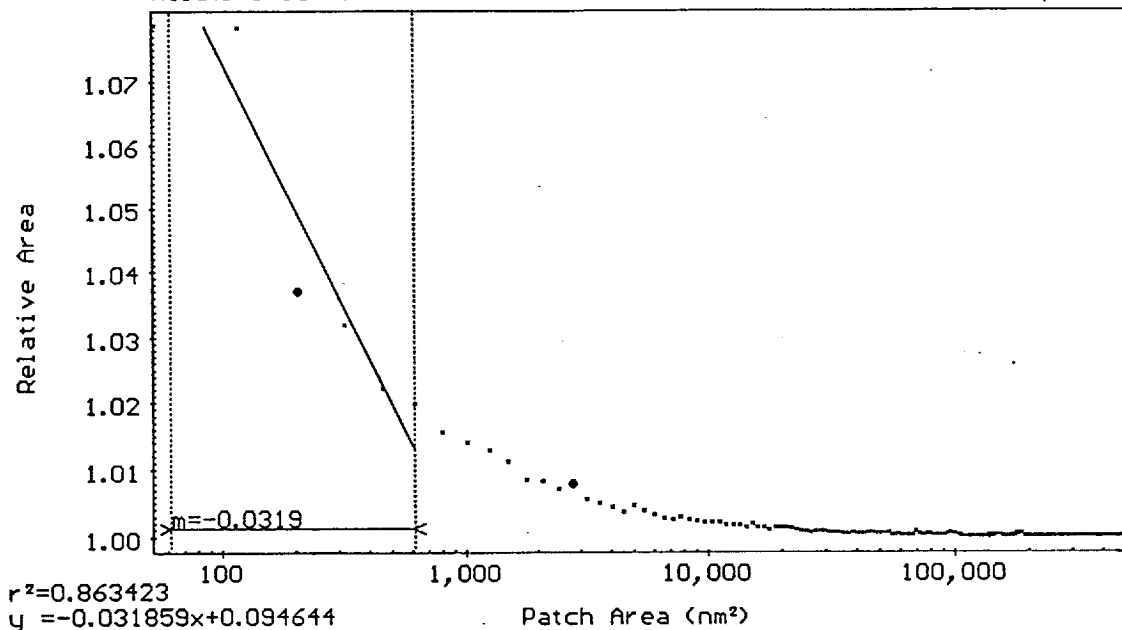


$r^2 = 0.819703$   
 $y = -0.000499x + 0.002329$   
 5.133e+03 to 5.133e+04

SOURCE - Copyright, Princeton U. Press  
 All Rights Reserved

\\data\\langley\\nasal1  
200 relative areas calculated  
Average/Patch Size & Surface Coverage  
Relative Surface Area vs. Patch Area

Quadrant (1,1) of (1,1)  
Crossover low: 205  
Crossover high: 2.81e+03  
Data-dependant



FORMAT - Langley, Dickinson & Brown  
ALL DATA ARE IN SI UNITS

\\data\\langley\\nasa12

200 relative areas calculated

Average/Patch Size & Surface Coverage

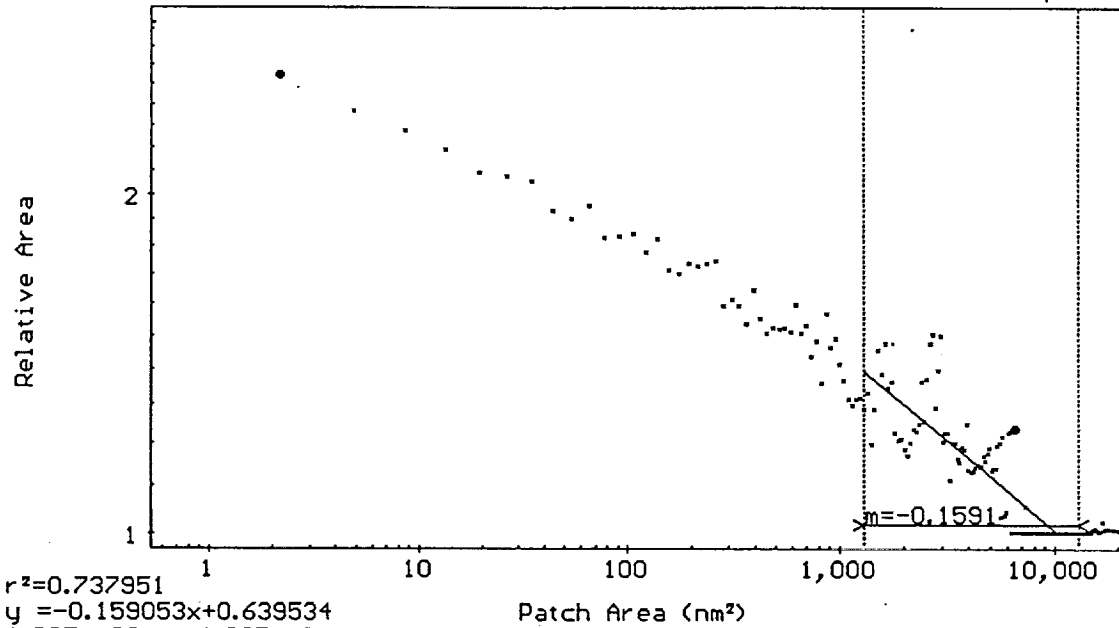
Relative Surface Area vs. Patch Area

Quadrant (1,1) of (1,1)

Crossover low: 2.16

Crossover high: 6.53e+03

Data-dependant



$r^2=0.737951$

$y = -0.159053x + 0.639534$

1.297e+03 to 1.297e+04

Patch Area (nm²)

NOTES - Example, Illustration & Data  
All Rights Reserved

\\data\\langley\\nasal3

200 relative areas calculated

Average/Patch Size & Surface Coverage

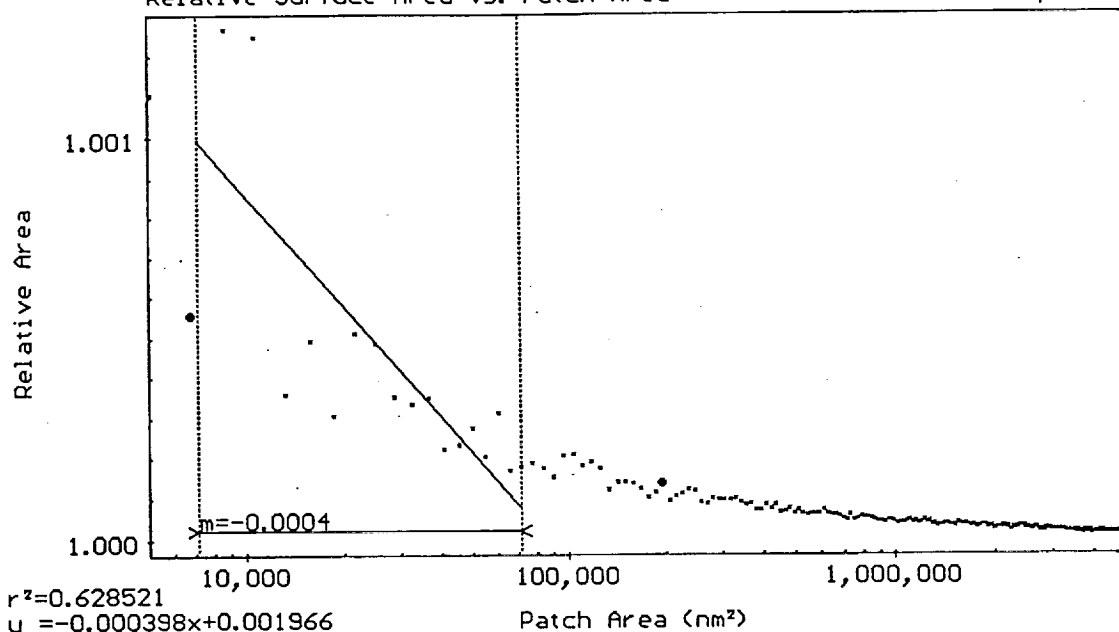
Relative Surface Area vs. Patch Area

Quadrant (1,1) of (1,1)

Crossover low: 6.76e+03

Crossover high: 1.95e+05

Data-dependant



$r^2 = 0.628521$

$y = -0.000398x + 0.001966$

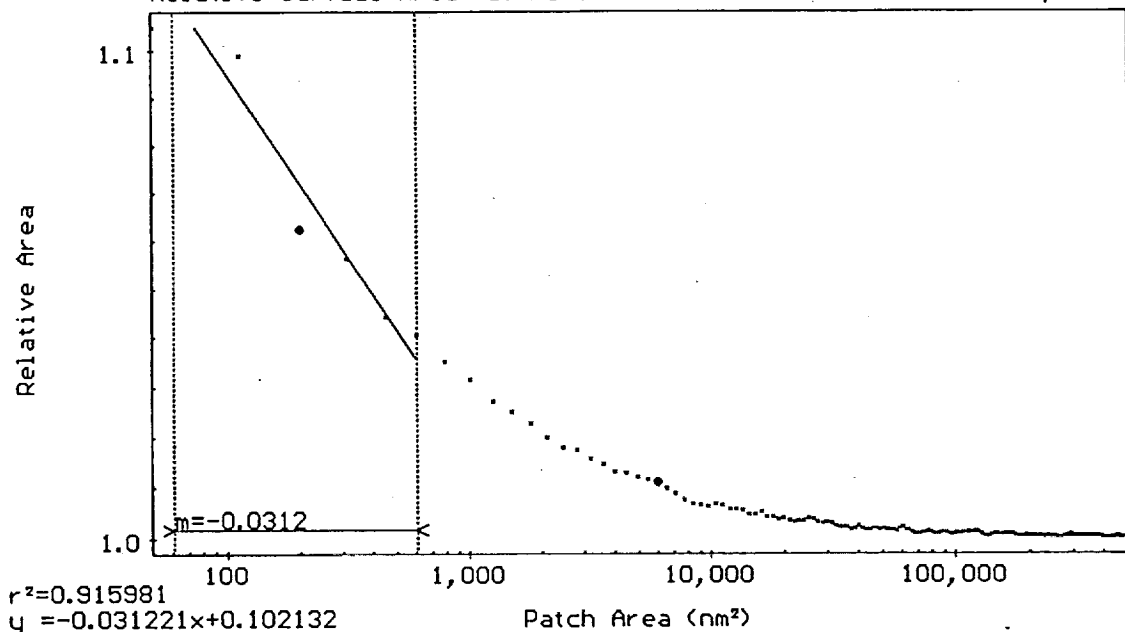
7.136e+03 to 7.136e+04

BMFLAC - Langley, Christopher A. Brown  
At 10:11:11 8/1/2013



\\data\\langley\\nasal4  
200 relative areas calculated  
Average/Patch Size & Surface Coverage  
Relative Surface Area vs. Patch Area

Quadrant (1,1) of (1,1)  
Crossover low: 200  
Crossover high: 6e+03  
Data-dependant



$r^2=0.915981$   
 $y=-0.031221x+0.102132$   
6.096e+01 to 6.096e+02

RESEARCH - Langley Research Center  
NASA

\\data\\langley\\nasa15

200 relative areas calculated

Average/Patch Size & Surface Coverage

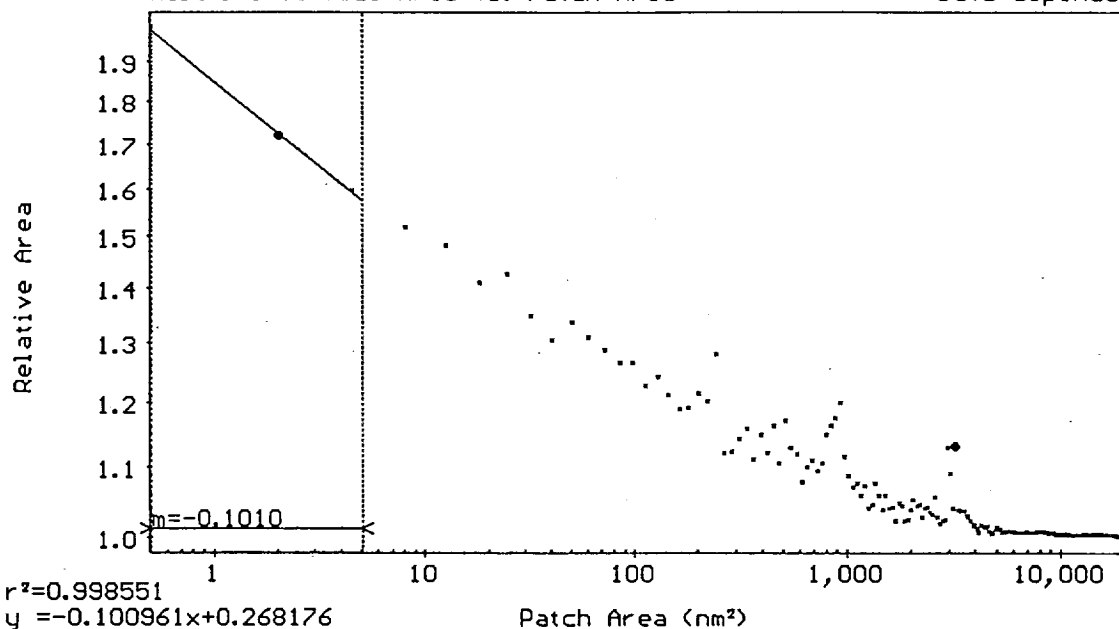
Relative Surface Area vs. Patch Area

Quadrant (1,1) of (1,1)

Crossover low: 2.02

Crossover high: 3.22e+03

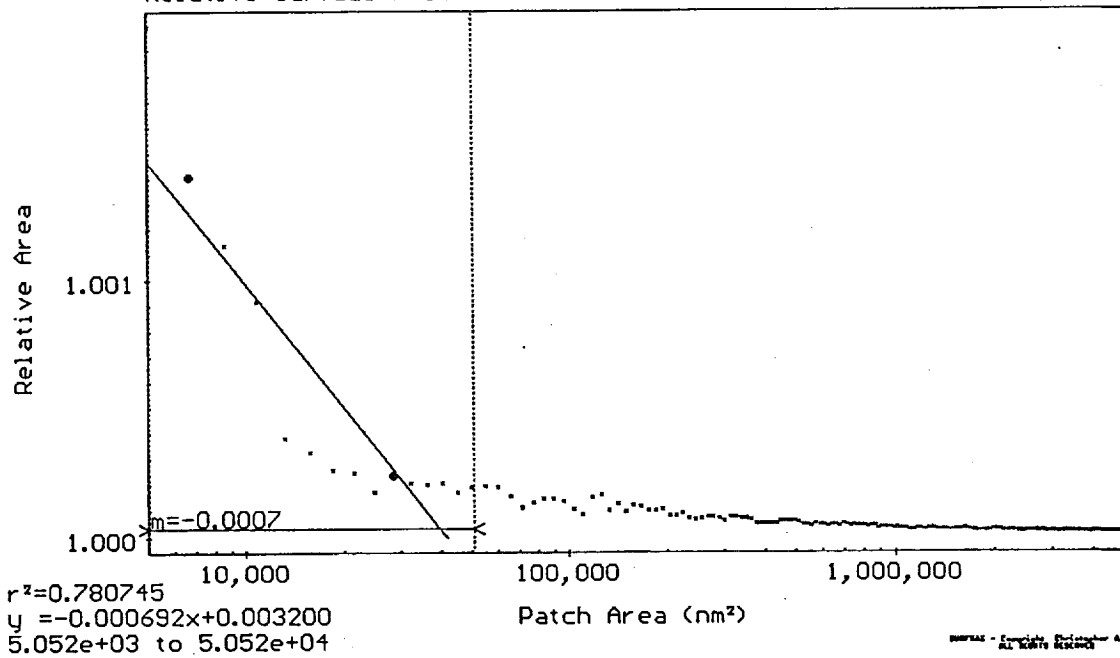
Data-dependant



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\\data\\langley\\nasal6  
200 relative areas calculated  
Average/Patch Size & Surface Coverage  
Relative Surface Area vs. Patch Area

Quadrant (1,1) of (1,1)  
Crossover low: 6.72e+03  
Crossover high: 2.86e+04  
Data-dependant



\\data\\langley\\nasa17

200 relative areas calculated

Average/Patch Size & Surface Coverage

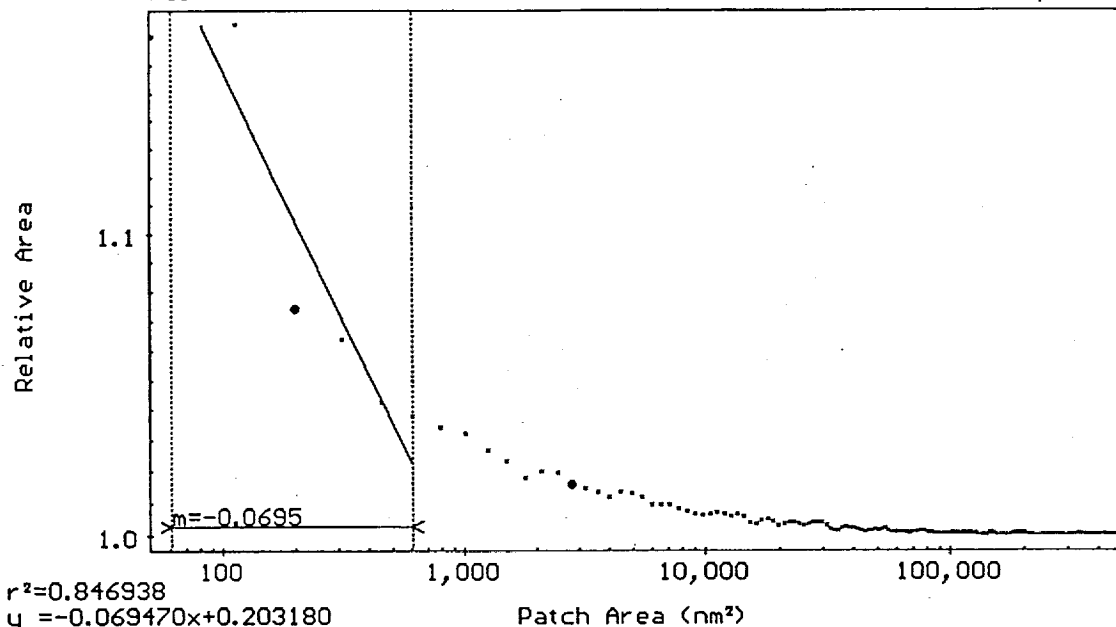
Relative Surface Area vs. Patch Area

Quadrant (1,1) of (1,1)

Crossover low: 200

Crossover high: 2.79e+03

Data-dependant



$r^2 = 0.846938$

$y = -0.069470x + 0.203180$

6.098e+01 to 6.098e+02

DATA - Langley, D. & Brown  
ALL DATA REPORT

\\data\\langley\\nasa18

200 relative areas calculated

Average/Patch Size & Surface Coverage

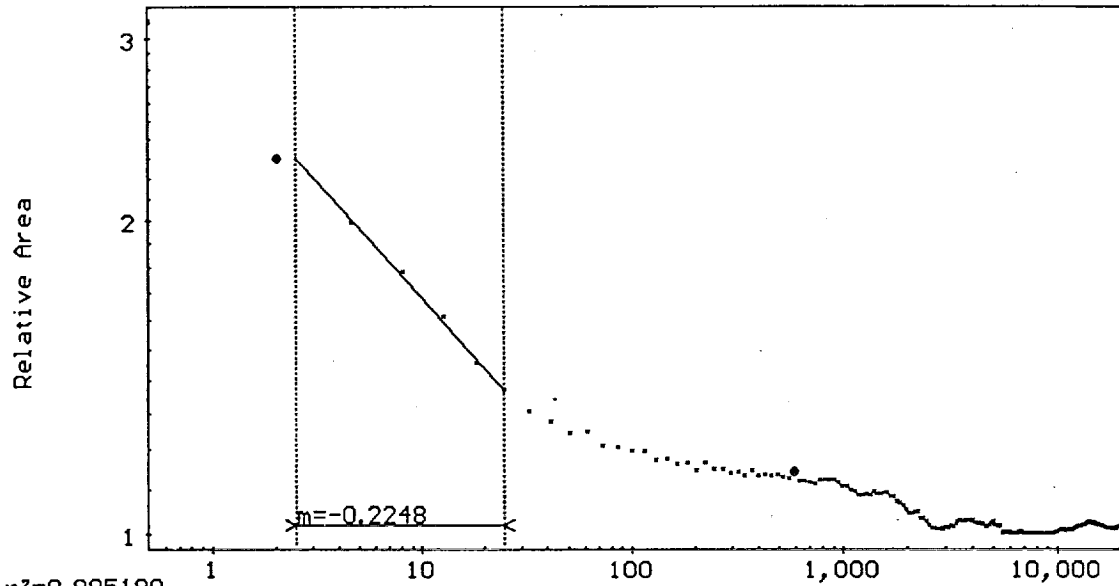
Relative Surface Area vs. Patch Area

Quadrant (1,1) of (1,1)

Crossover low: 2.04

Crossover high: 590

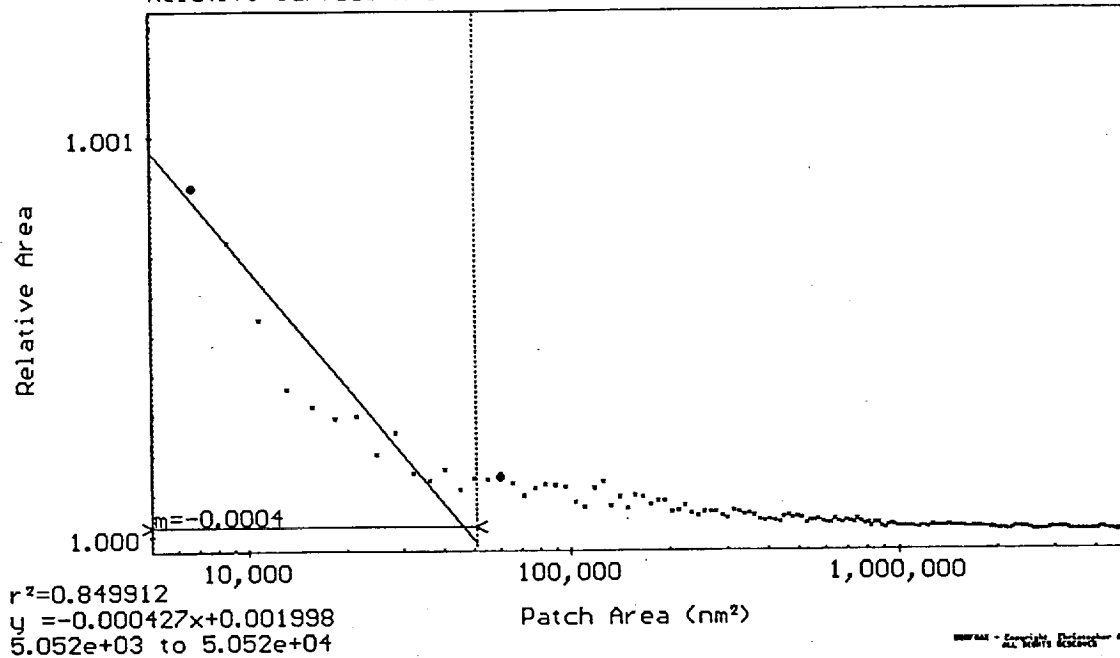
Data-dependant



SWTAK - Example, Quadrant 1, 1  
22 March 1993

\\data\\langley\\nasa19  
200 relative areas calculated  
Average/Patch Size & Surface Coverage  
Relative Surface Area vs. Patch Area

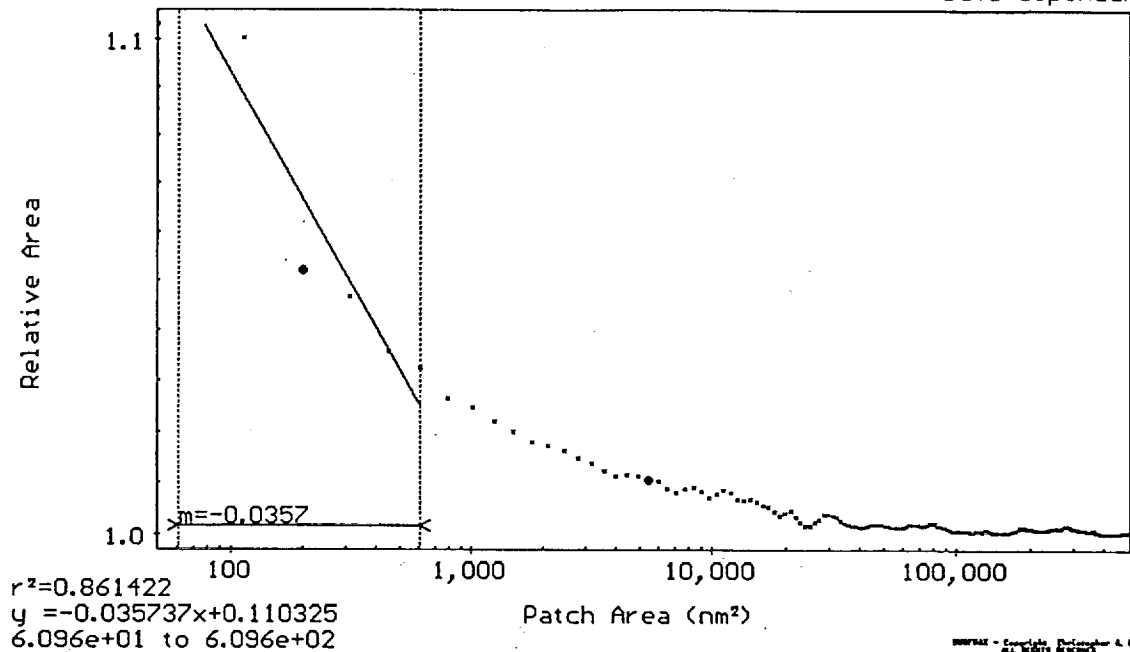
Quadrant (1,1) of (1,1)  
Crossover low:  $6.72 \times 10^3$   
Crossover high:  $6.01 \times 10^4$   
Data-dependant



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\data\langley\nasa20  
 200 relative areas calculated  
 Average/Patch Size & Surface Coverage  
 Relative Surface Area vs. Patch Area

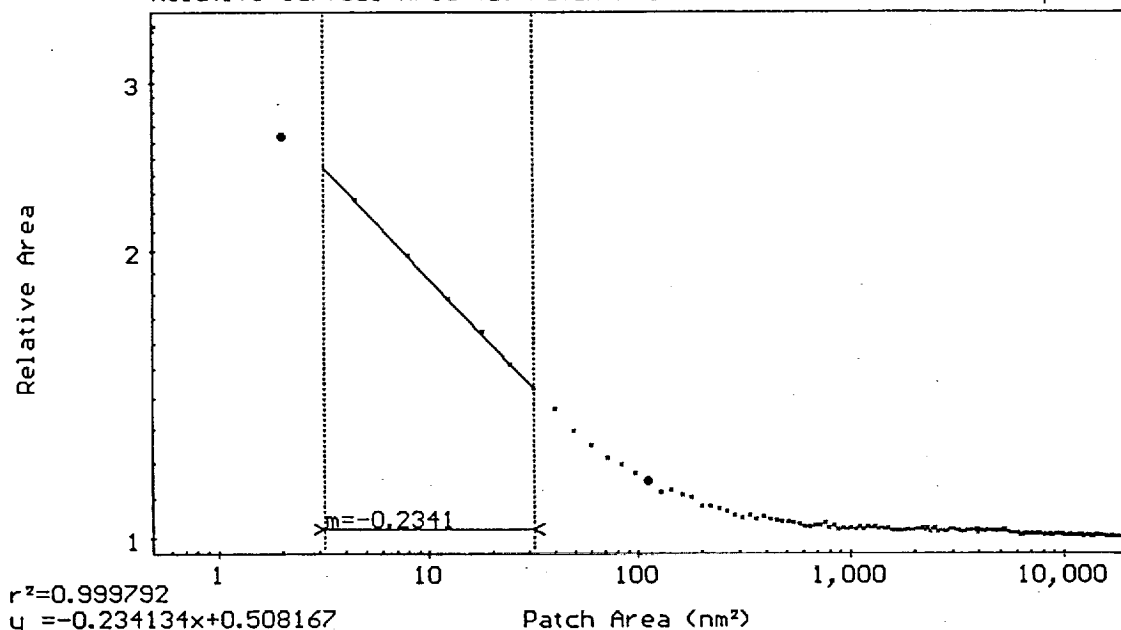
Quadrant (1,1) of (1,1)  
 Crossover low: 200  
 Crossover high: 5.47e+03  
 Data-dependant



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\data\langley\nasa21  
 200 relative areas calculated  
 Average/Patch Size & Surface Coverage  
 Relative Surface Area vs. Patch Area

Quadrant (1,1) of (1,1)  
 Crossover low: 2  
 Crossover high: 112  
 Data-dependant



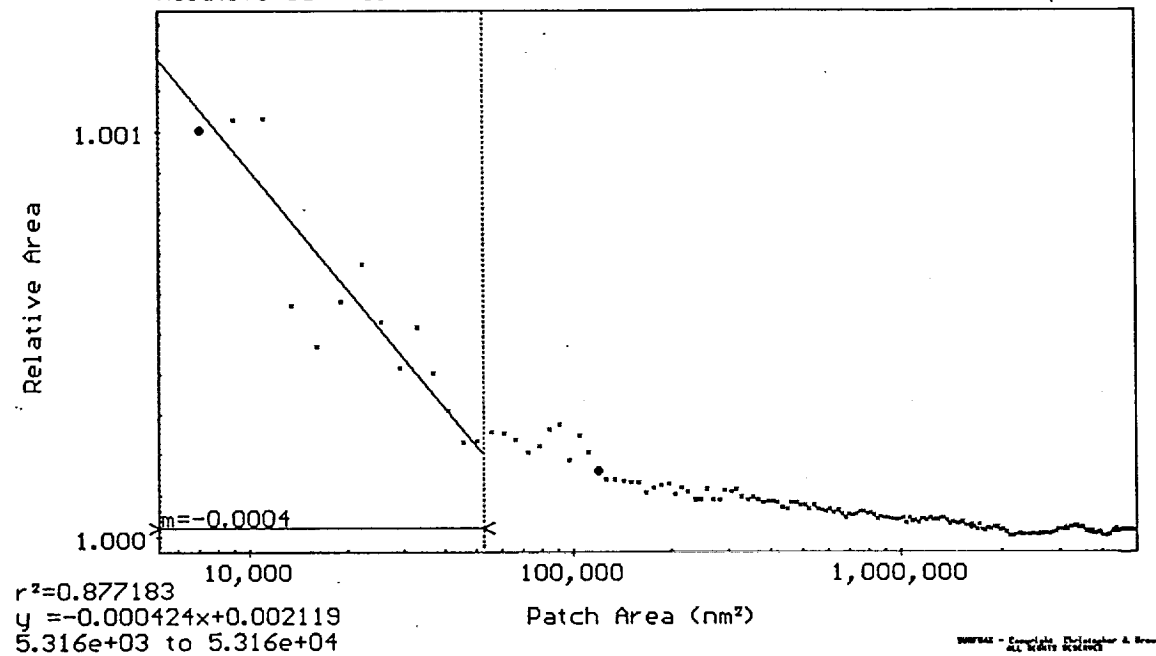
$r^2 = 0.999792$   
 $C = -0.234134x + 0.508167$   
 3.181e+00 to 3.181e+01

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\data\langley\nasa22  
 200 relative areas calculated  
 Average/Patch Size & Surface Coverage  
 Relative Surface Area vs. Patch Area

Quadrant (1,1) of (1,1)  
 Crossover low: 7.02e+03  
 Crossover high: 1.19e+05  
 Data-dependant



\\data\\langley\\nasa23

200 relative areas calculated

Average/Patch Size & Surface Coverage

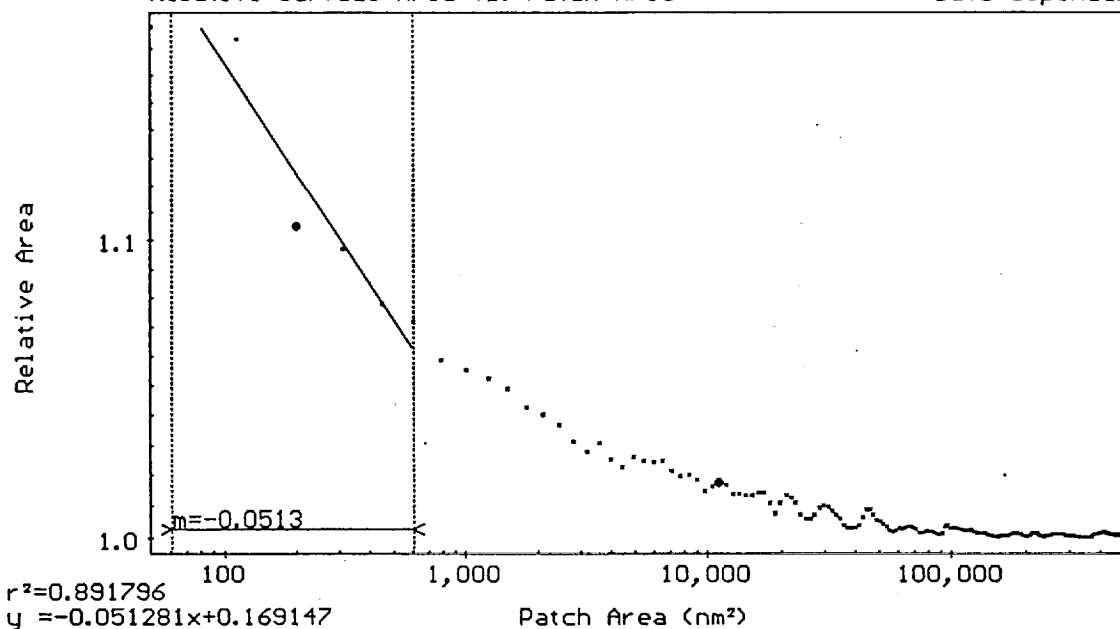
Relative Surface Area vs. Patch Area

Quadrant (1,1) of (1,1)

Crossover low: 200

Crossover high: 1.12e+04

Data-dependant



$r^2 = 0.891796$

$y = -0.051281x + 0.169147$

6.096e+01 to 6.096e+02

NSA/CSS - Security, Privileged & Confidential  
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\\data\\langley\\nasa24

200 relative areas calculated

Average/Patch Size & Surface Coverage

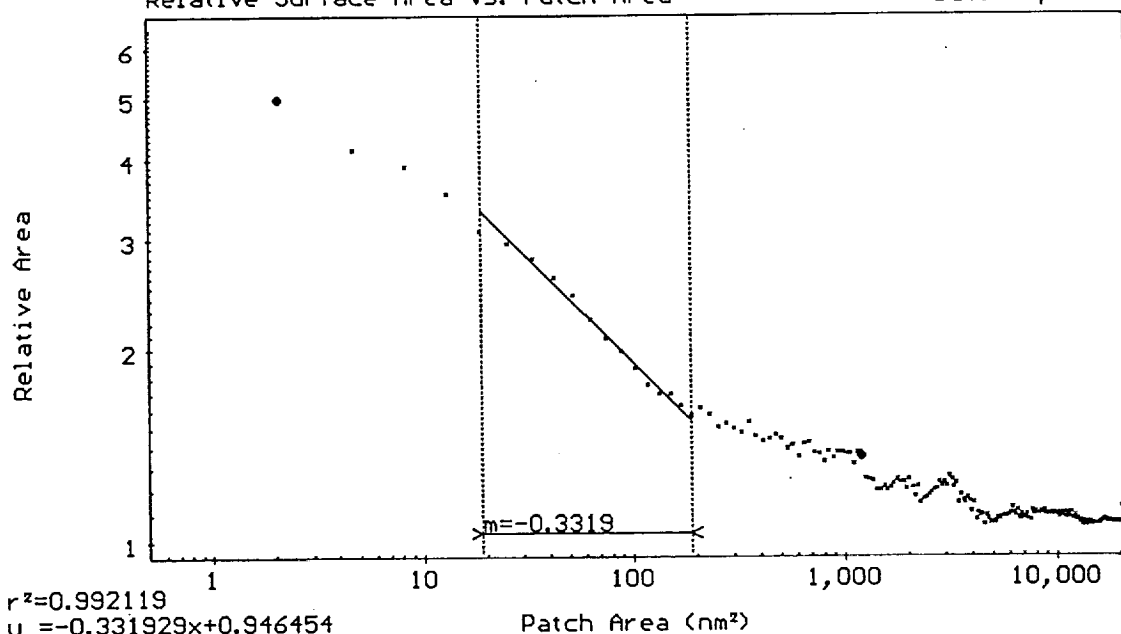
Relative Surface Area vs. Patch Area

Quadrant (1,1) of (1,1)

Crossover low: 2.06

Crossover high: 1.21e+03

Data-dependent



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\\data\\langley\\nasa25

200 relative areas calculated

Average/Patch Size & Surface Coverage

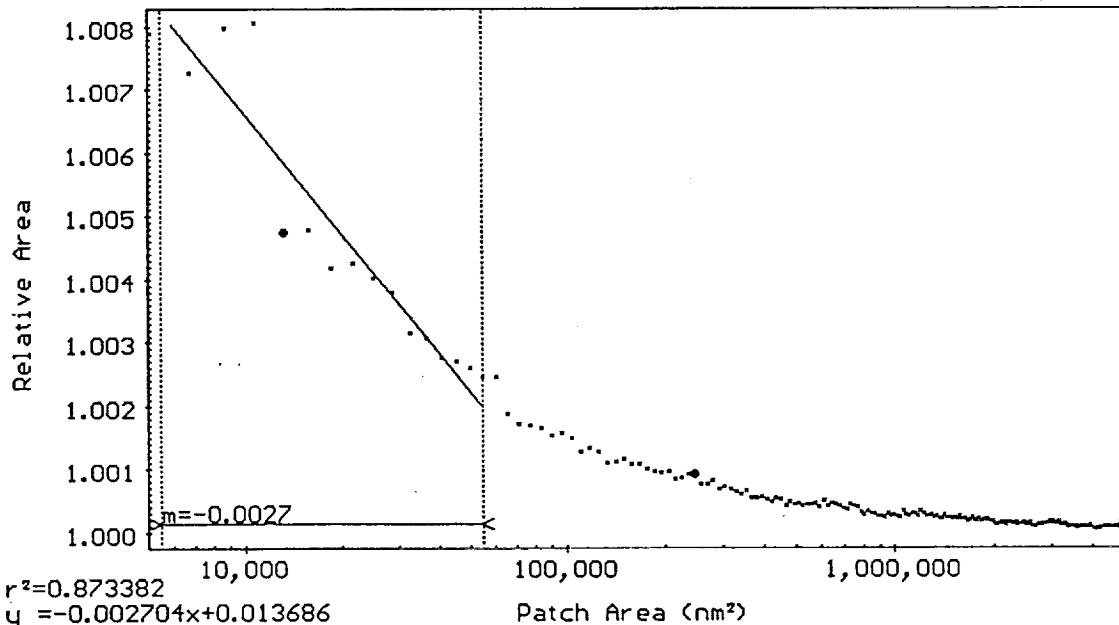
Relative Surface Area vs. Patch Area

Quadrant (1,1) of (1,1)

Crossover low: 1.32e+04

Crossover high: 2.46e+05

Data-dependant



$r^2 = 0.873382$

$y = -0.002704x + 0.013686$

5.499e+03 to 5.499e+04

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ALL RIGHTS RESERVED

\\data\\langley\\nasa26

200 relative areas calculated

Average/Patch Size & Surface Coverage

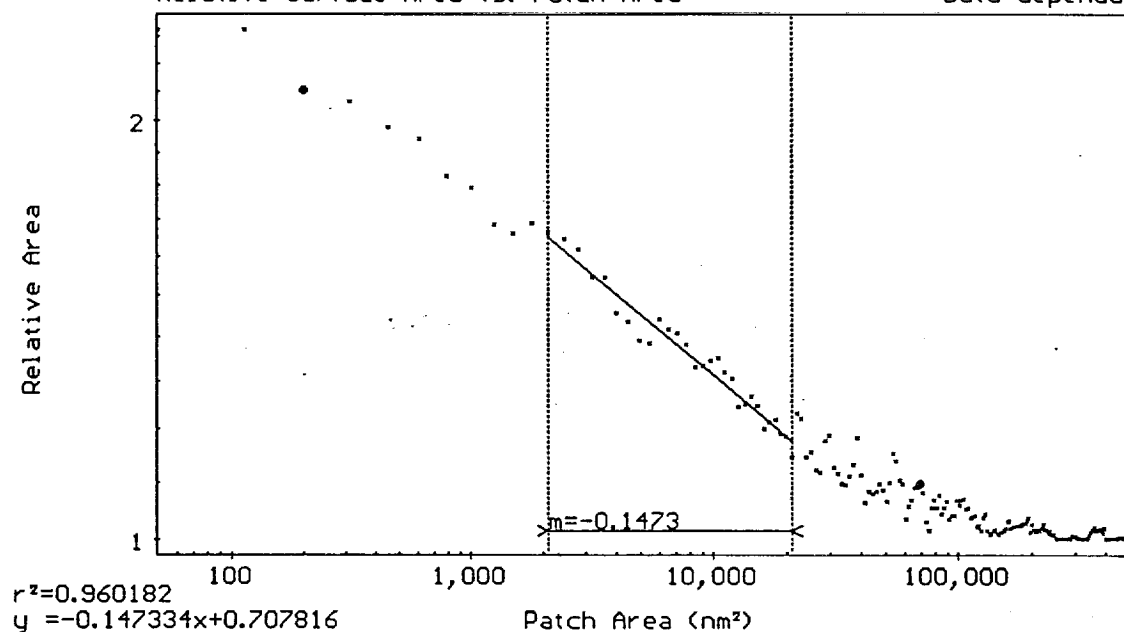
Relative Surface Area vs. Patch Area

Quadrant (1,1) of (1,1)

Crossover low: 200

Crossover high: 6.96e+04

Data-dependant



DATA - Langley, R. J. & G. S. 1991

\\data\\langley\\nasa27

200 relative areas calculated

Average/Patch Size & Surface Coverage

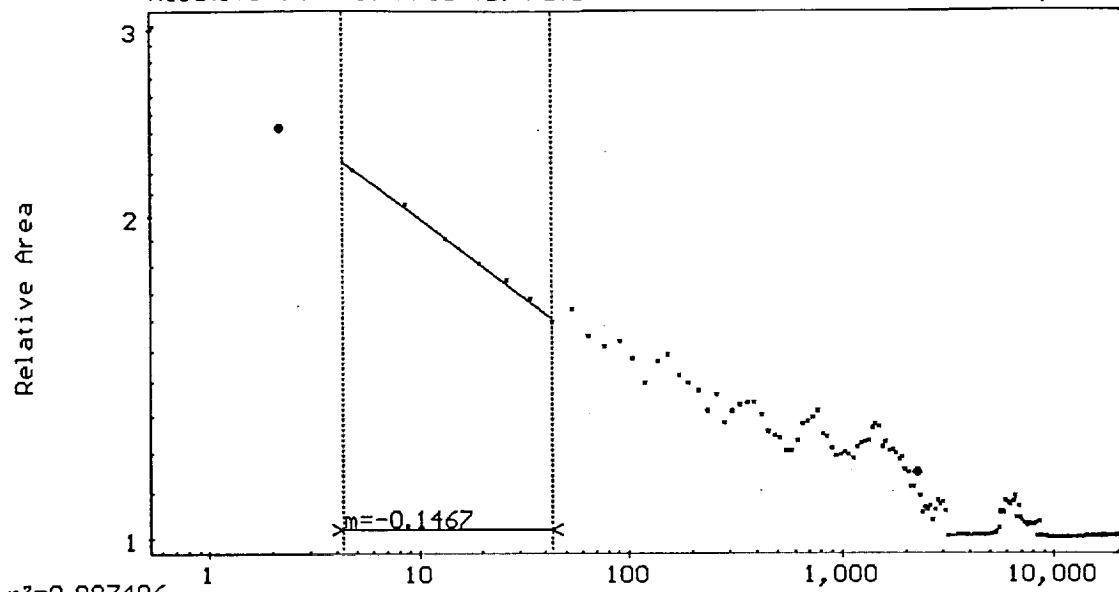
Relative Surface Area vs. Patch Area

Quadrant (1,1) of (1,1)

Crossover low: 2.15

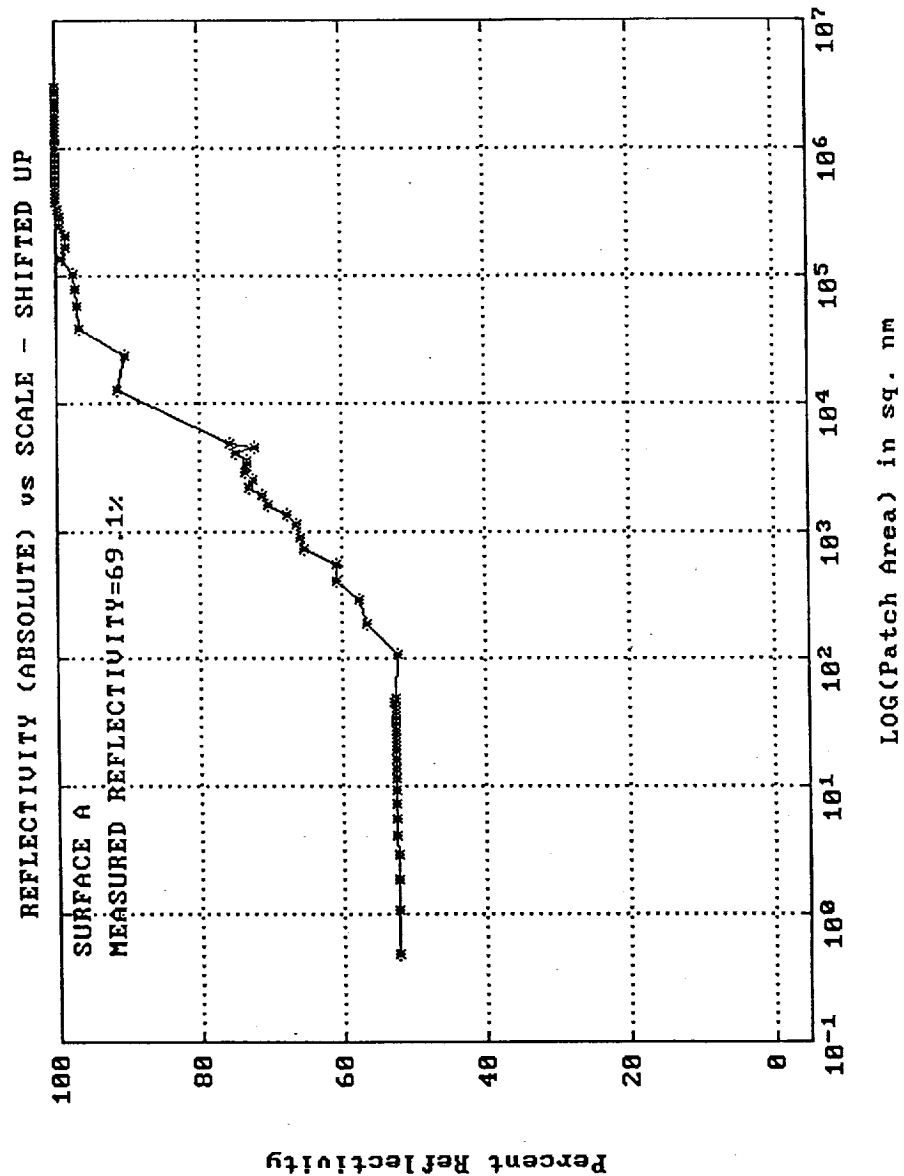
Crossover high: 2.26e+03

Data-dependant

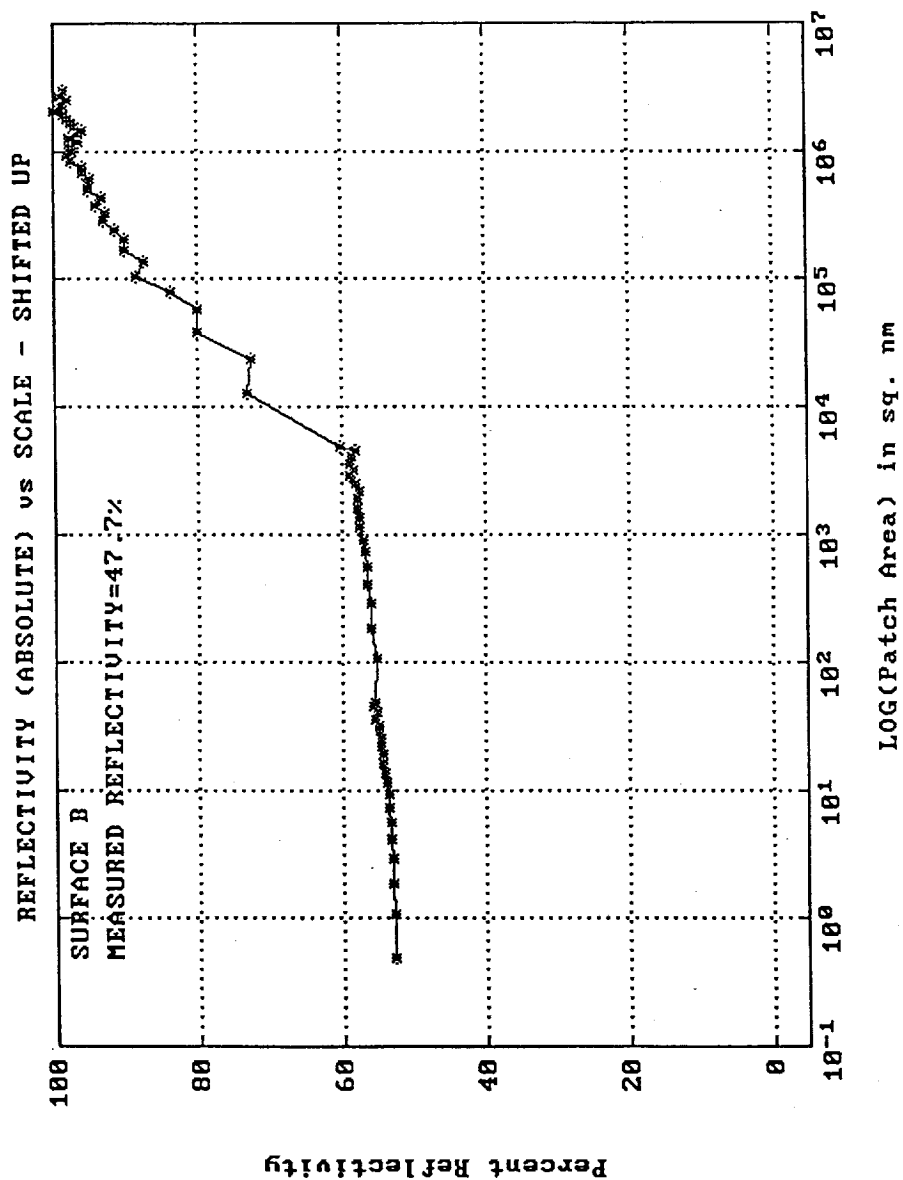


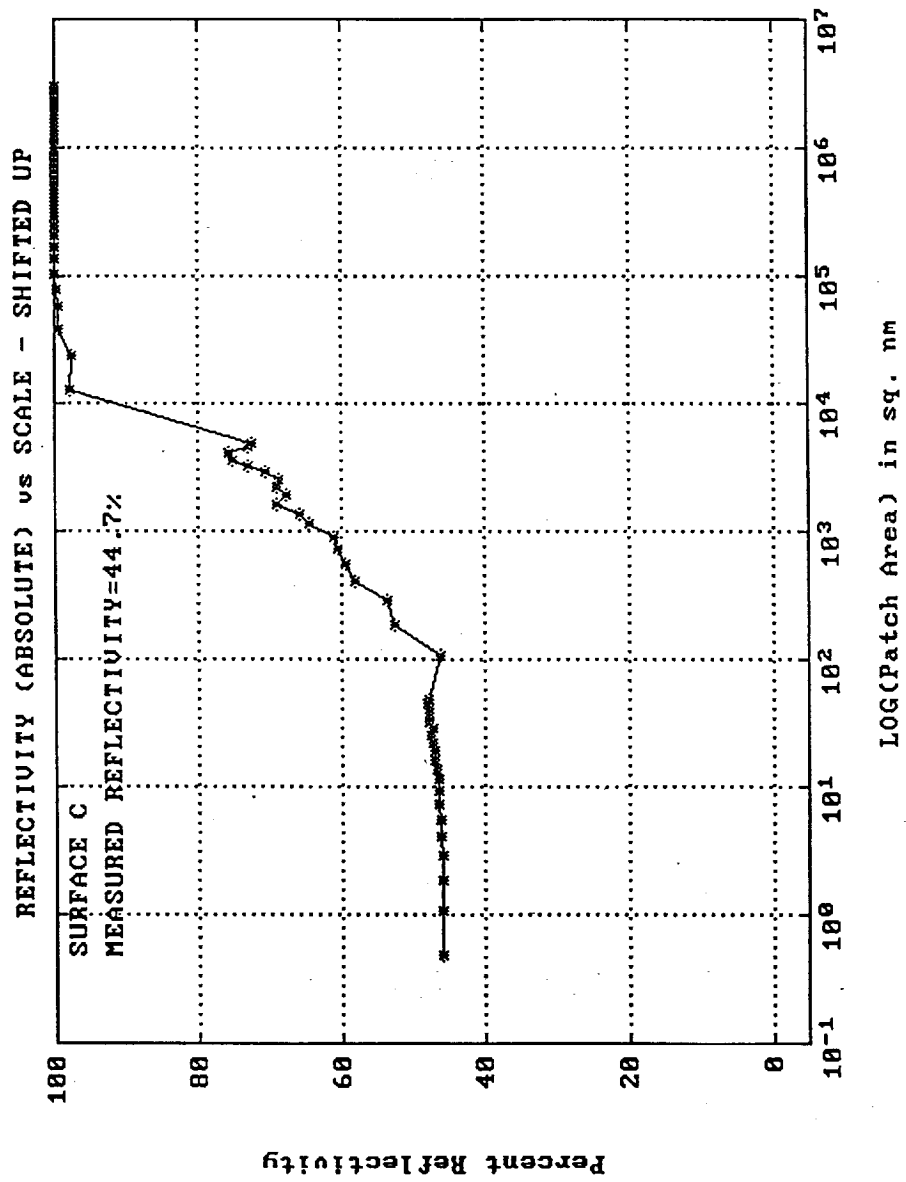
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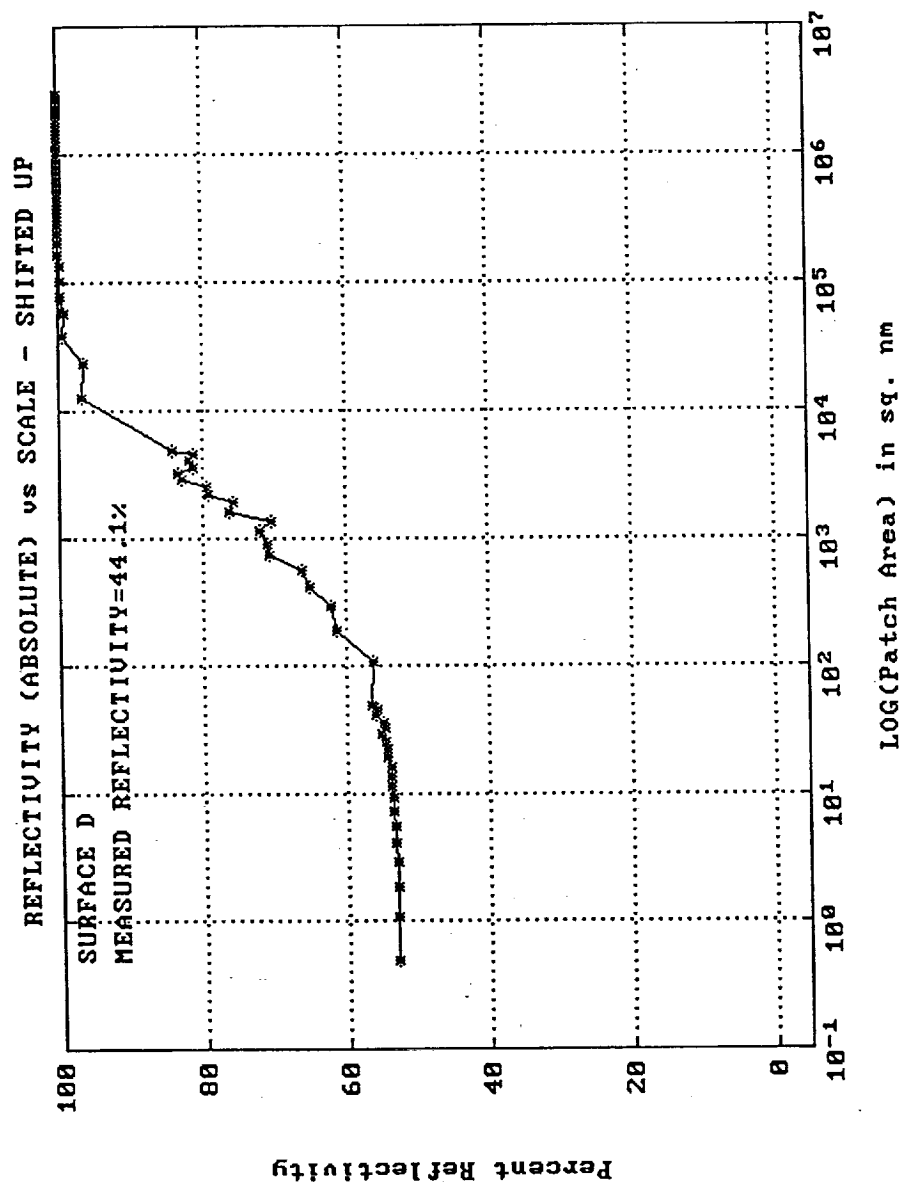
**Appendix B**  
**Absolute Reflectivity vs. Patch Area**

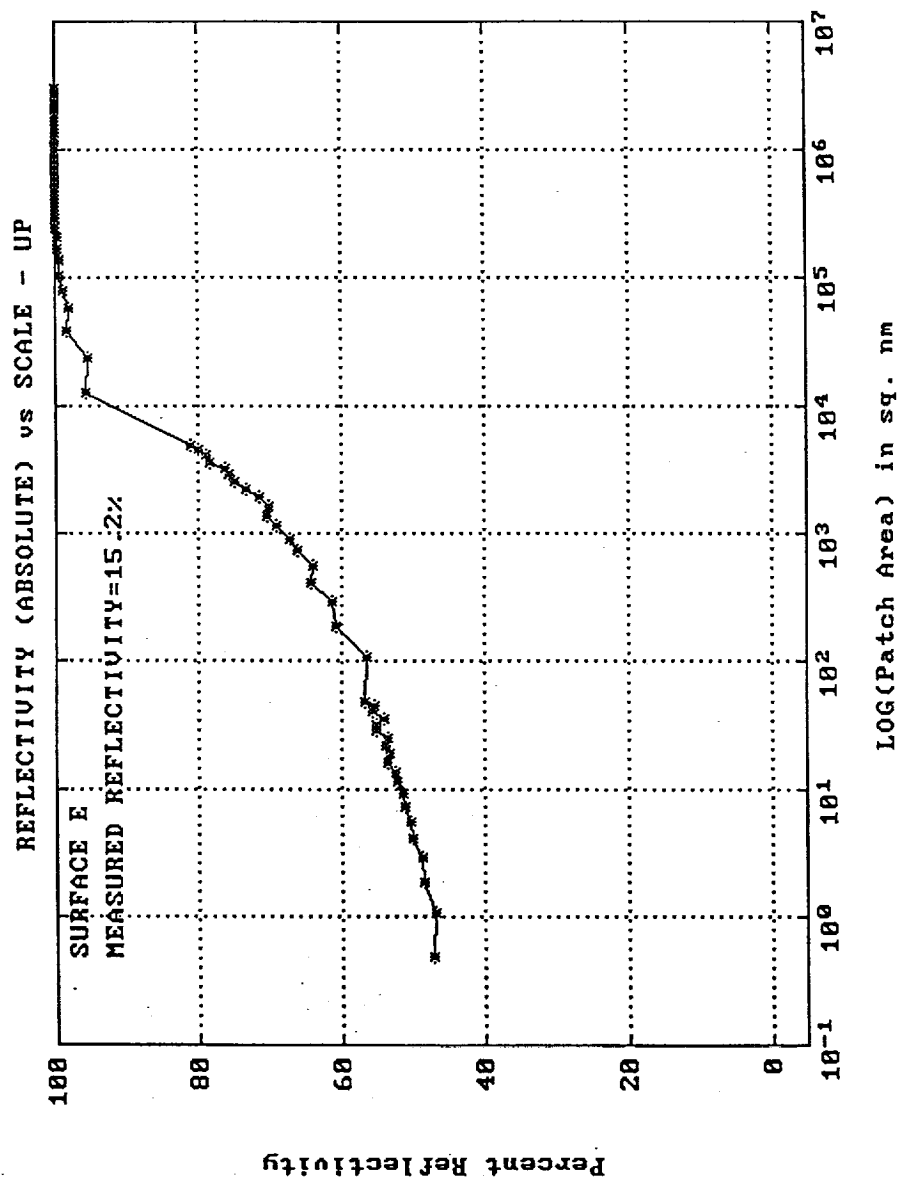


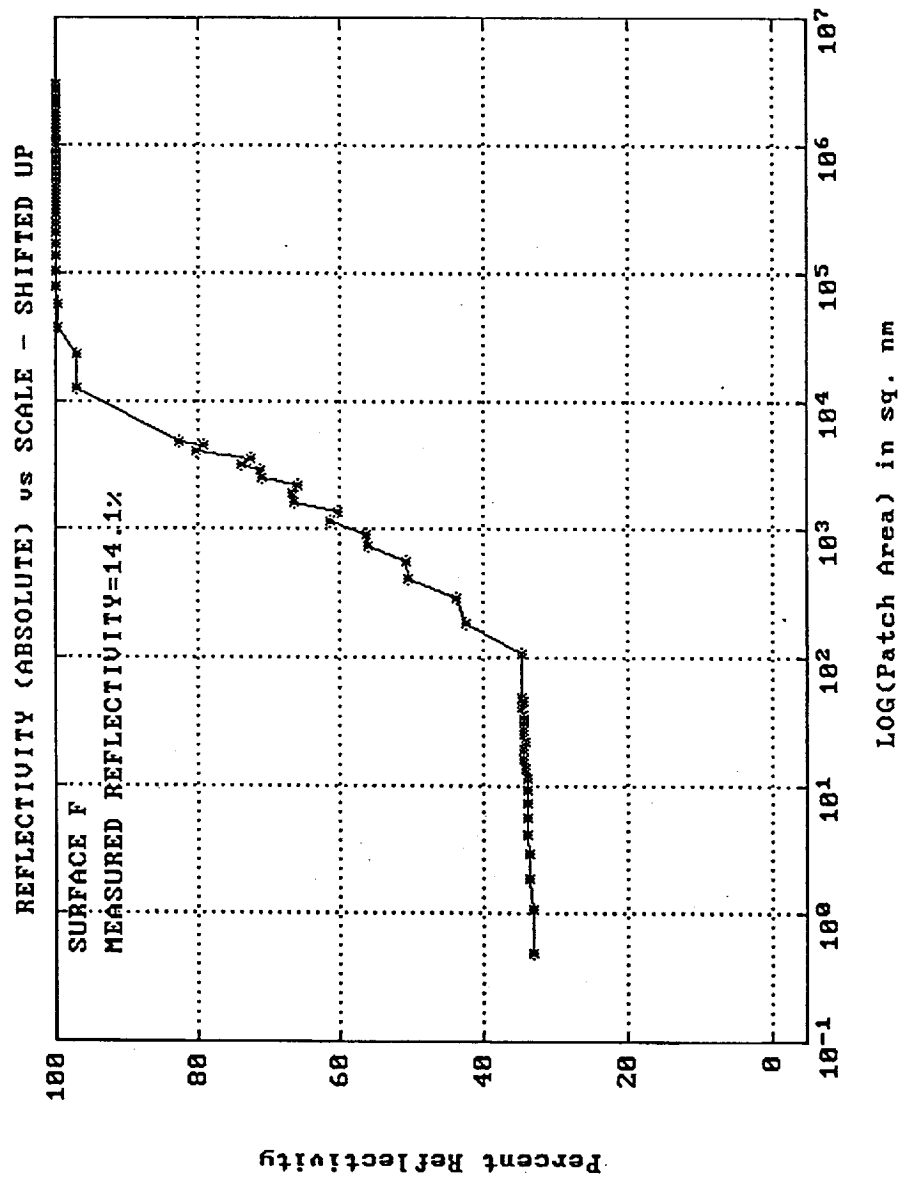


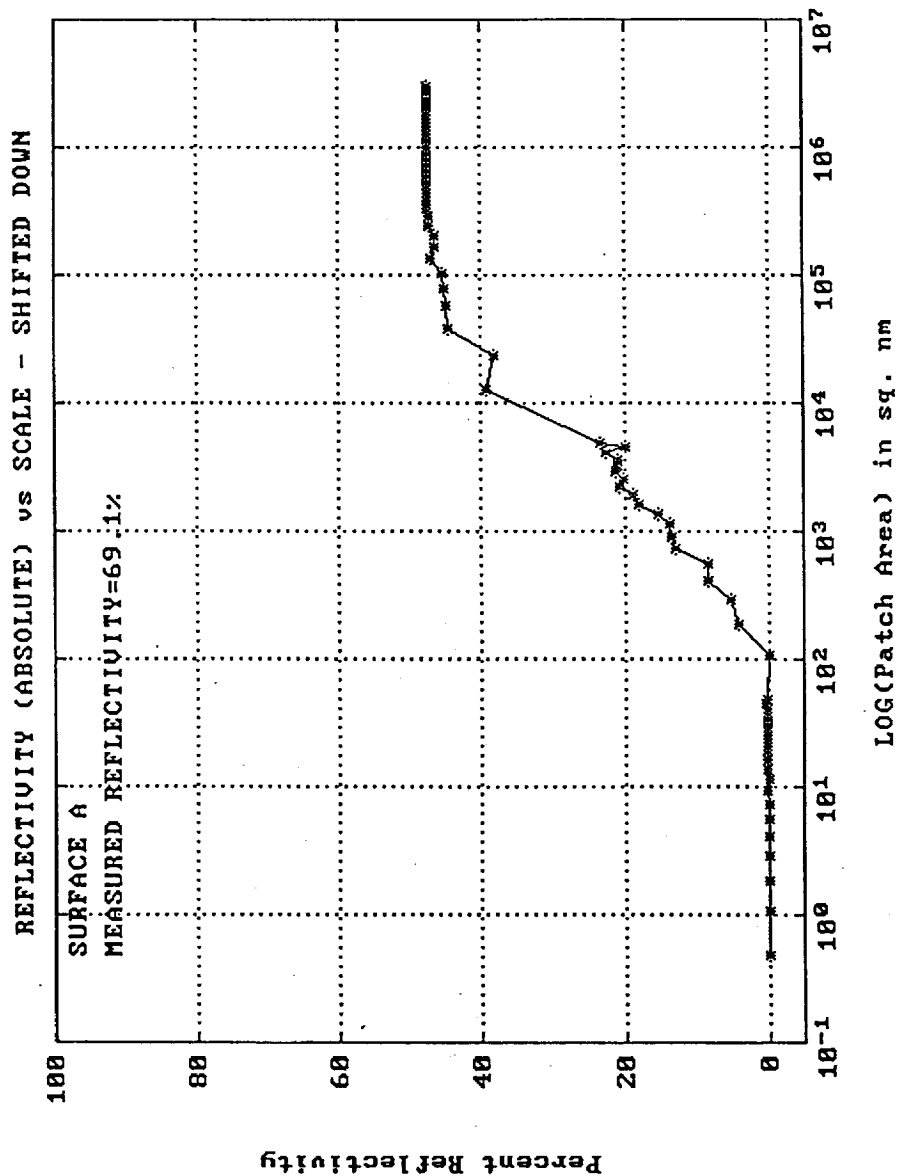








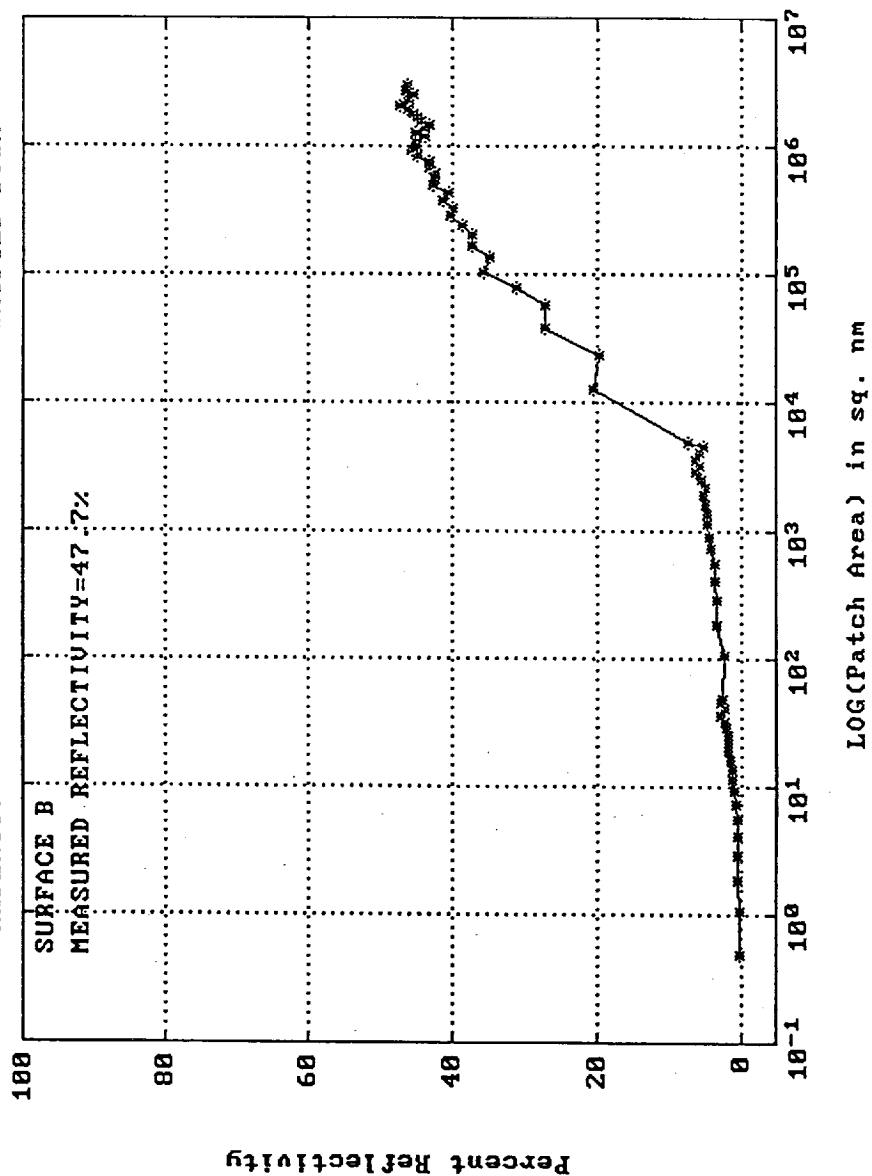


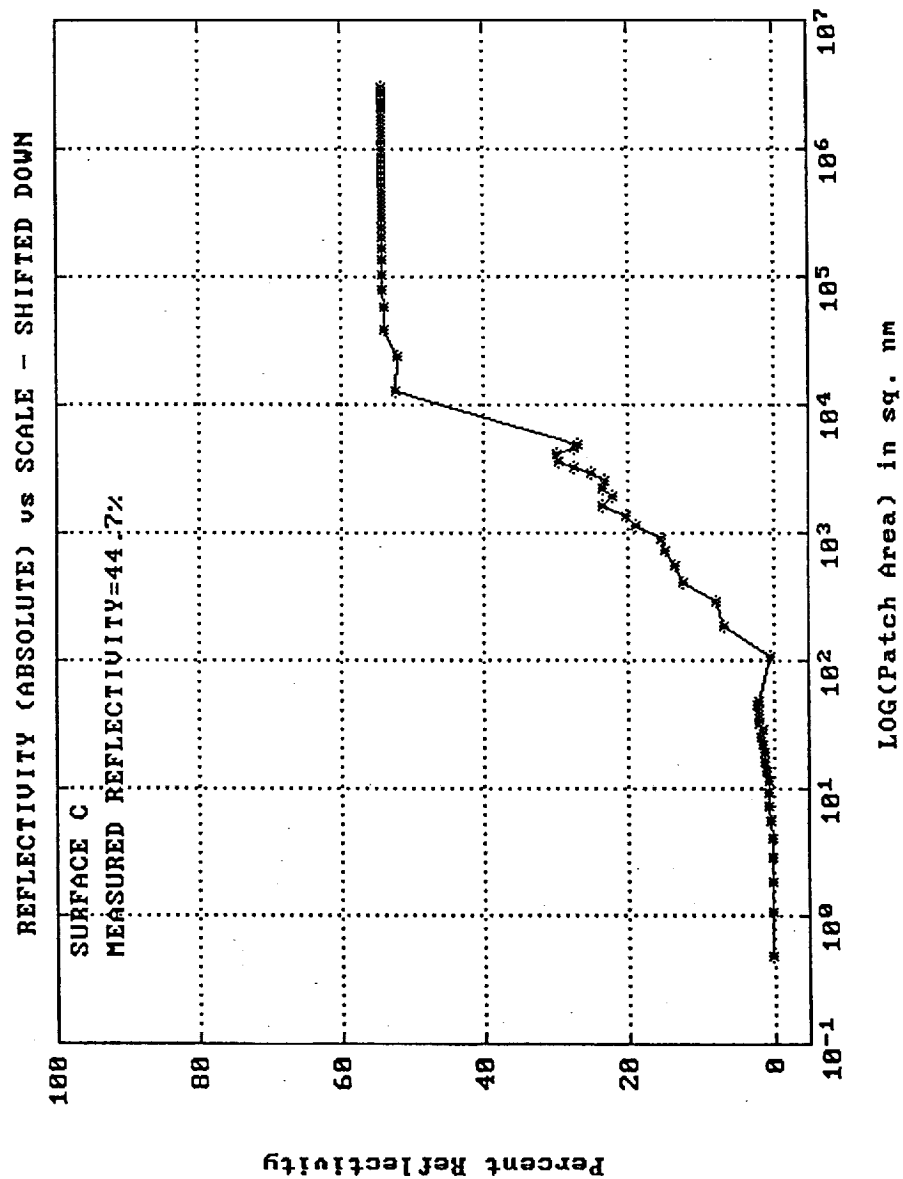


REFLECTIVITY (ABSOLUTE) vs SCALE - SHIFTED DOWN

SURFACE B

MEASURED REFLECTIVITY=47.7%



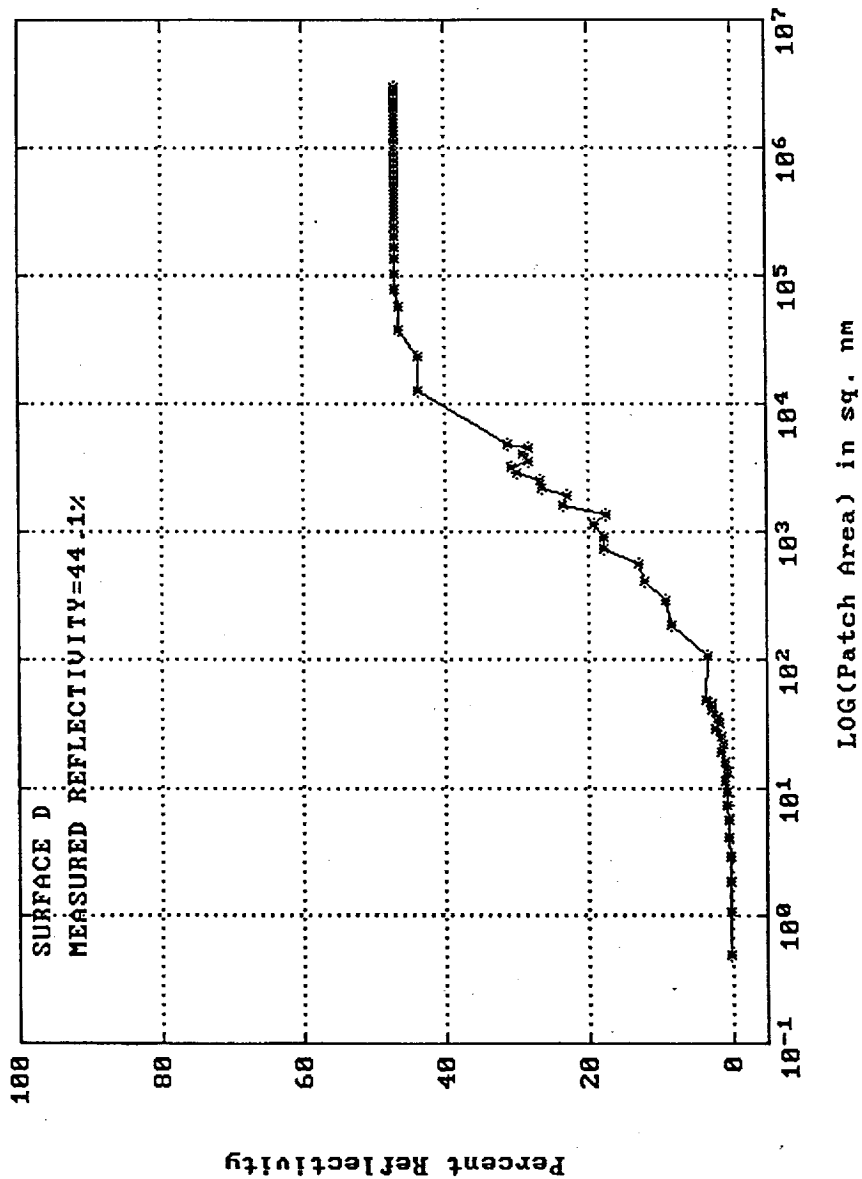


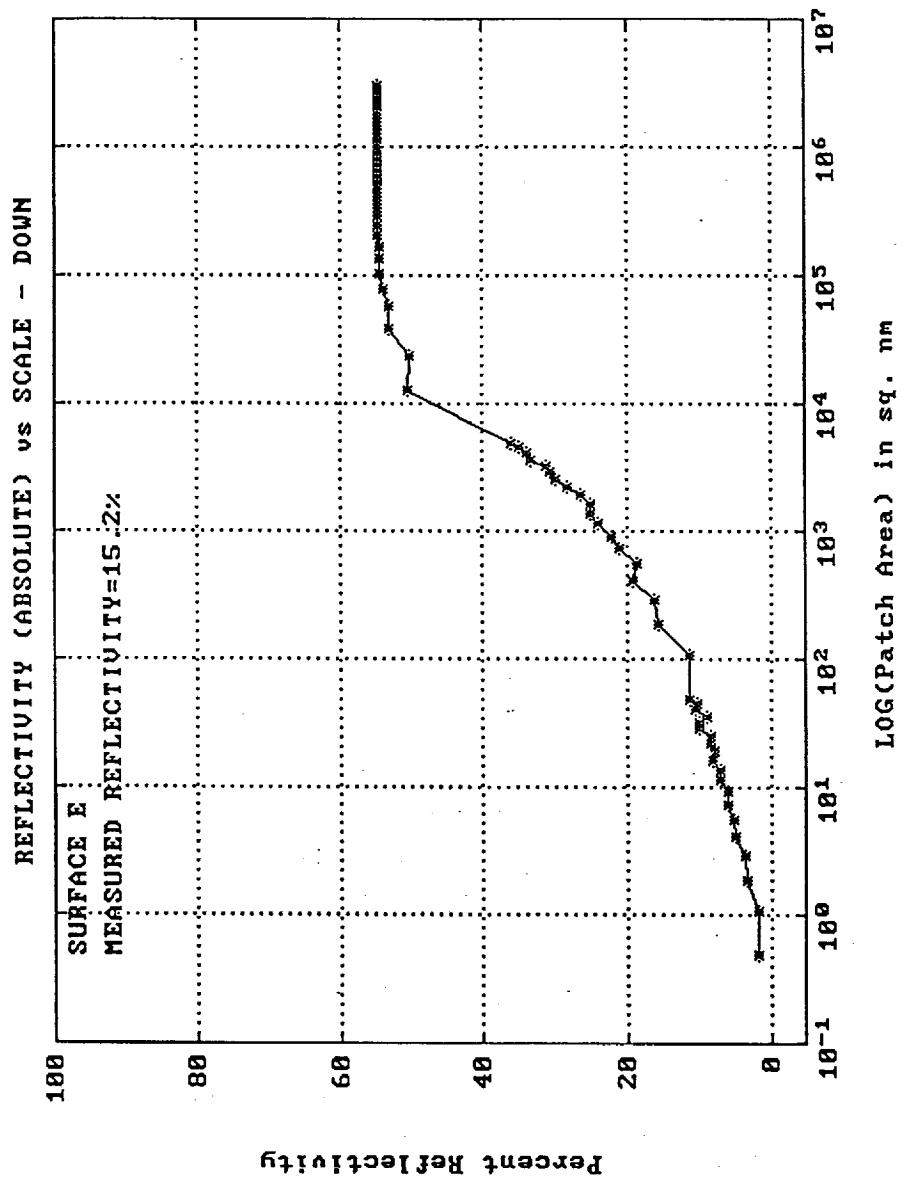


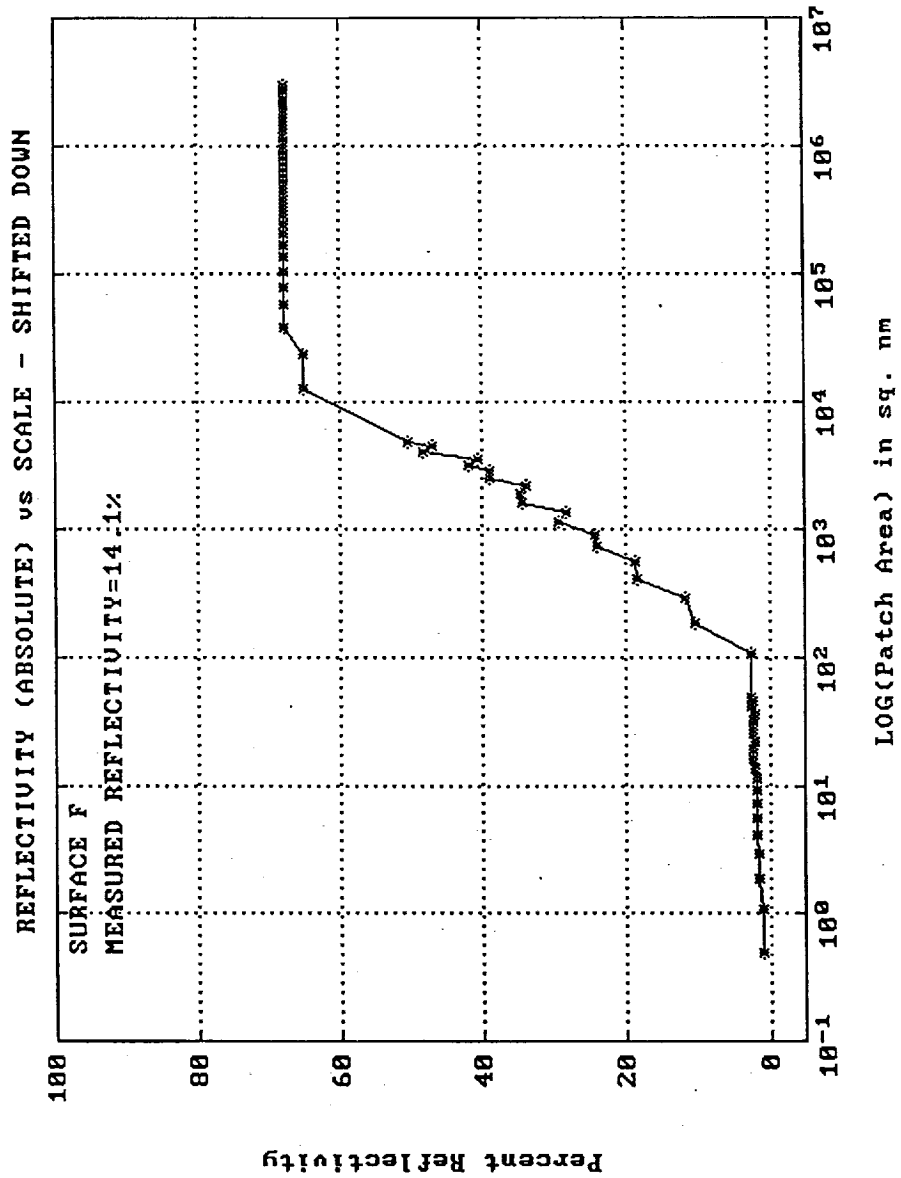
REFLECTIVITY (ABSOLUTE) vs SCALE - SHIFTED DOWN

SURFACE D

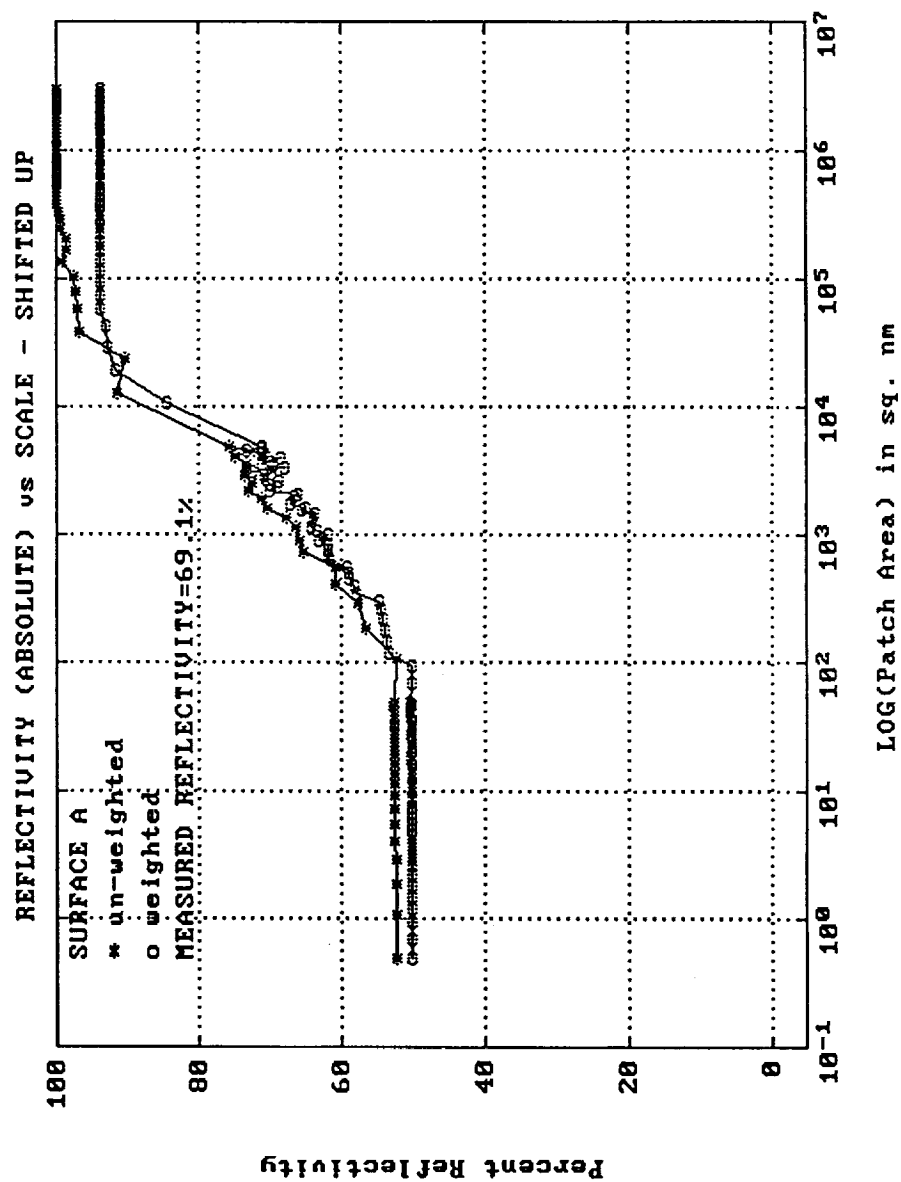
MEASURED REFLECTIVITY=44.1%

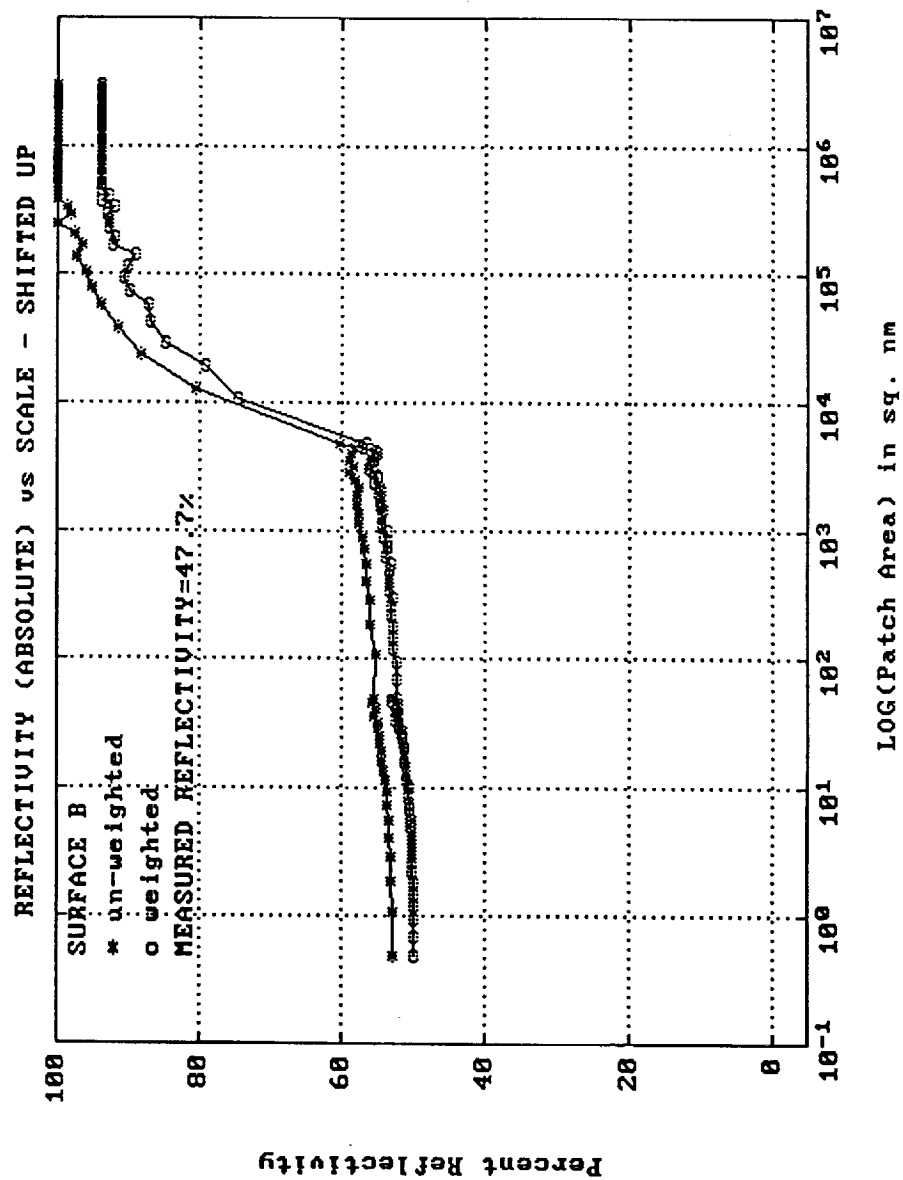


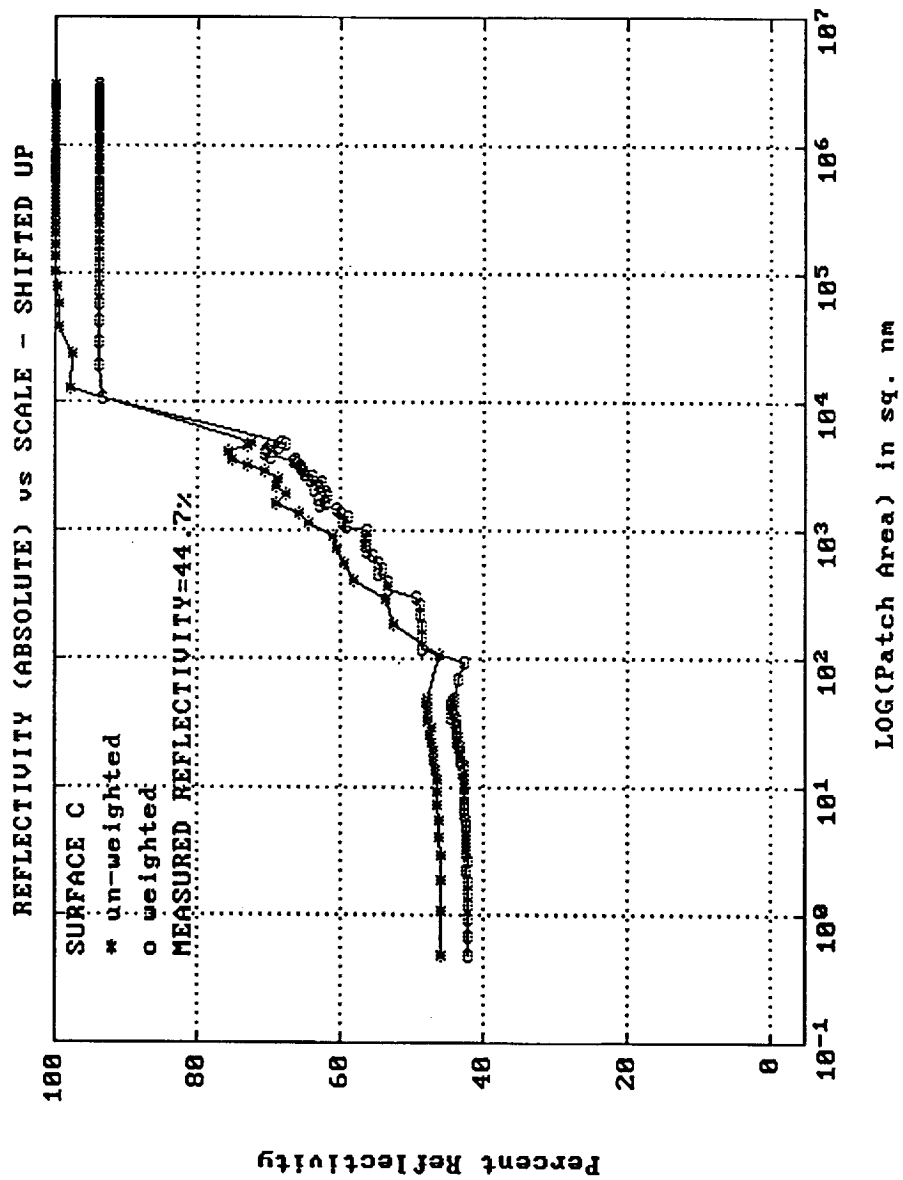


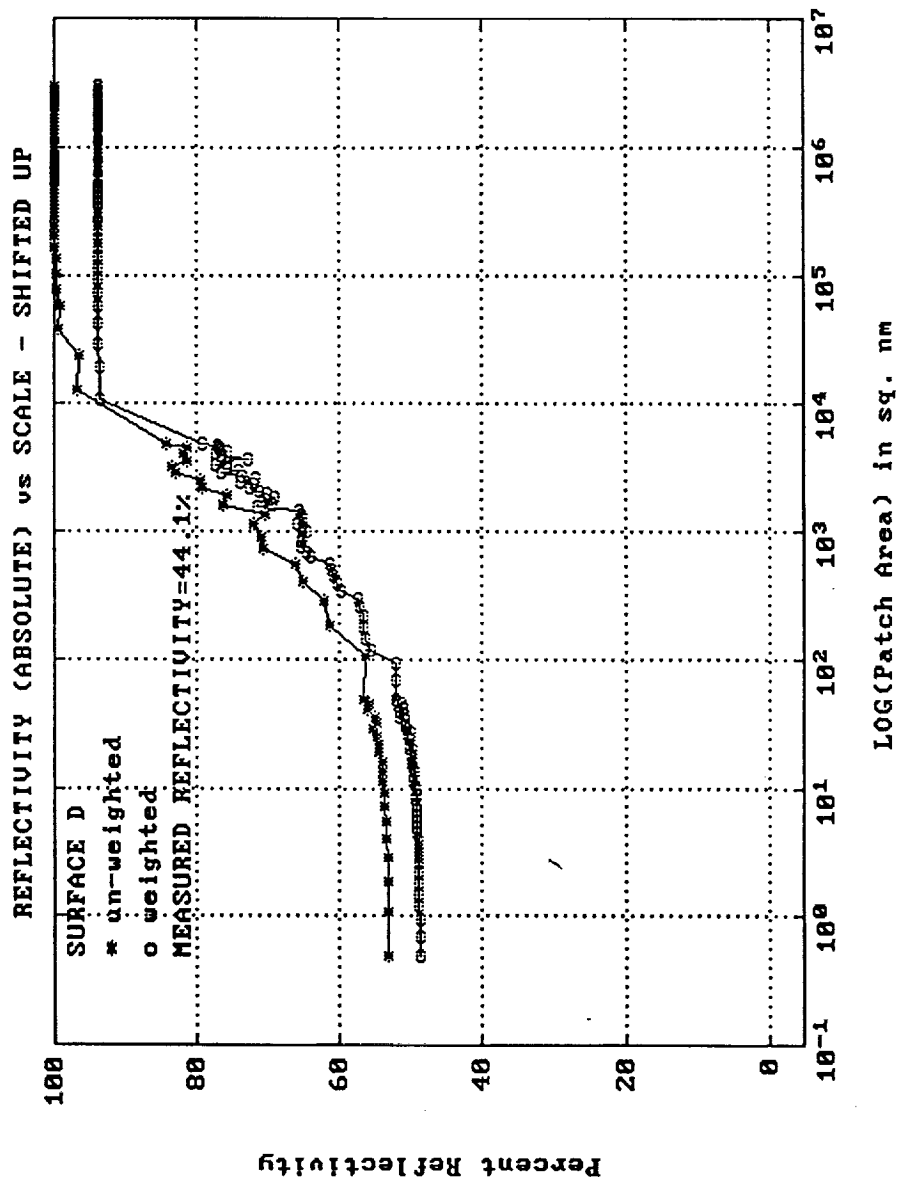


**Appendix C**  
**Cosine Weighted and Non-Weighted Absolute Reflectivity vs Patch Area**

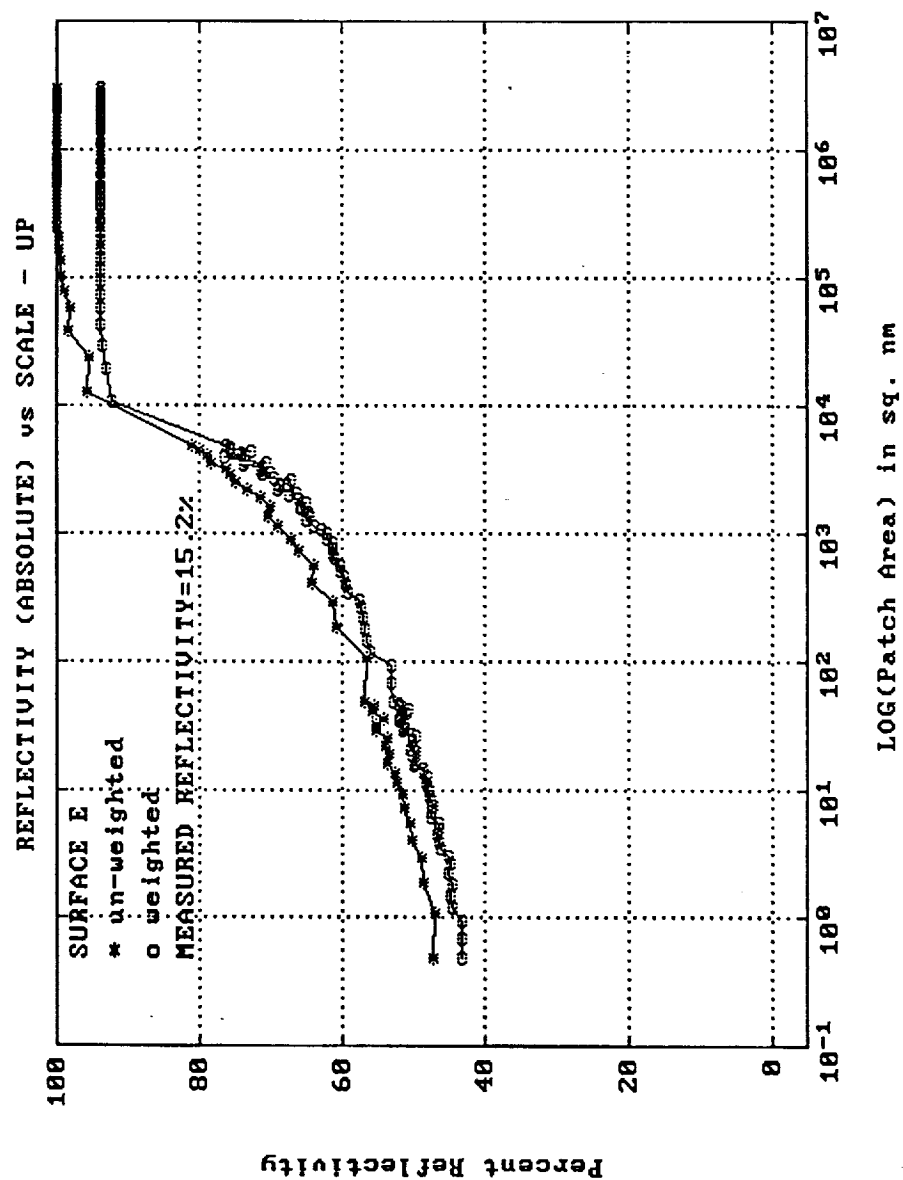


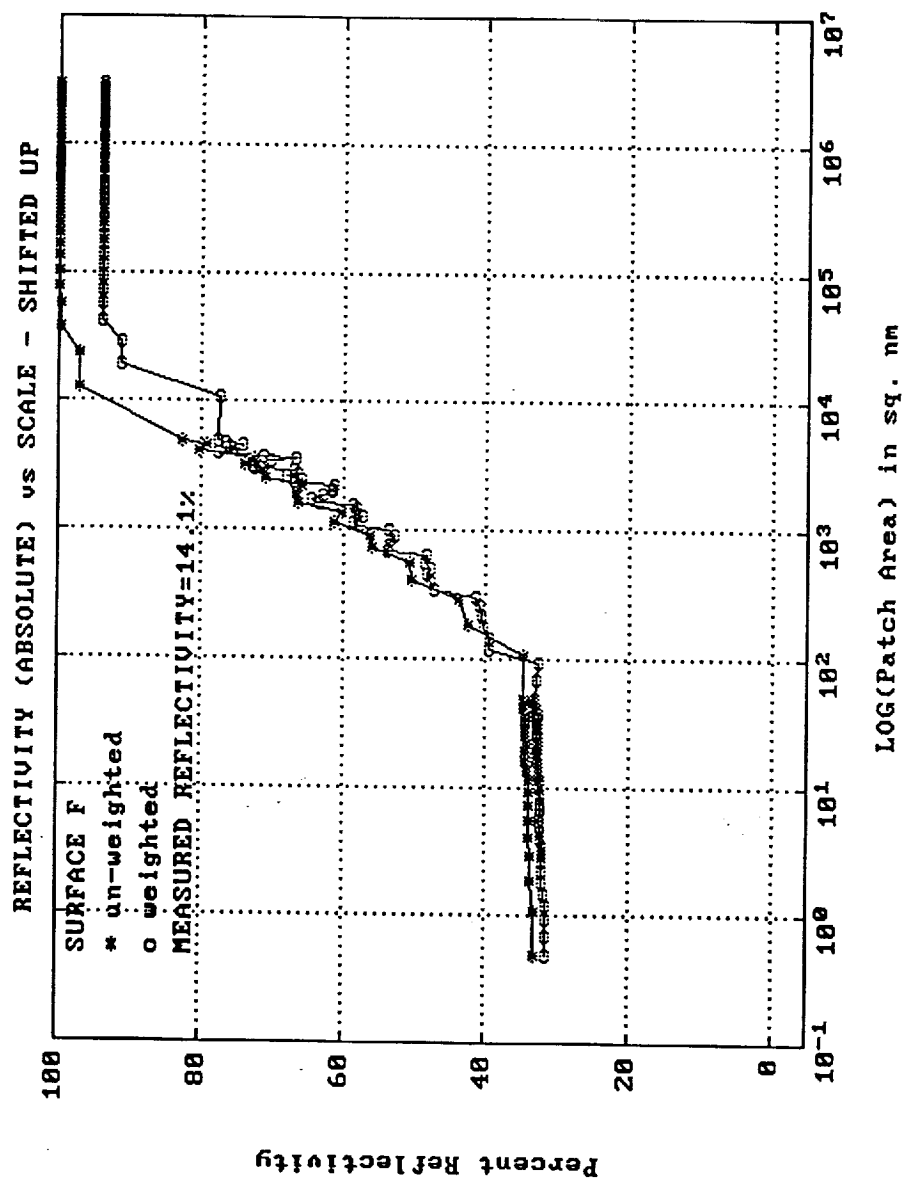












**Appendix D**  
**Relative Reflectivity vs. Patch Area**

